INVESTIGATION OF SUBSTITUTES FOR COBALT IN WC-CO ALLOYS AND EFFECT OF GRAIN SIZE ON THE PROPERTIES OF WC-CO ALLOYS

(FINAL REPORT)

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15 June 1955

Submitted by:

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FINAL TECHNICAL REPORT

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Contract Number:	DA-19-020-ORD-3410.
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WAL File Number:	330/13
Date:	15 June 1955
Title of Project:	Investigation of Substitute for Cobalt in WC-Co alloys and Effect of Grain Size on the Properties of WC-Co Alloys.

Object:

To find a suitable substitute for Co in WC-Co alloys which would produce compacts possessing densities greater than 13.6 g/cc. and Rockwell "A" hardnesses greater than 85. Also, to study the effects of grain size on the properties of WC-Co alloys.

Summary:

Five metallic elements and three metallic alloys were investigated as possible substitutes for cobalt in cemented wolfram (tungsten) carbide. Hardnesses, specific gravities, porosities and grain sizes were measured for compacts hot pressed from wolfram carbide powder containing binders other than cobalt. Compression strengths were measured on compacts made from wolfram carbide and each of several promising substitute binders. Compacts were prepared from finely powdered wolfram carbide and cobalt in such a manner that the finished compacts differed in grain size. The effect of grain size was determined on hardness, specific gravity and compression strength.

CONCLUSIONS:

1. Wolfram(tungsten) carbide iron compacts have been produced by hot pressing methods with compressive strengths and hardnesses equal to WC-Co compacts. In view of the highly strategic nature of cobalt, more work on this promising development should be done.

2. Alloy binders of nickel and iron, or nickel and copper with WC, also offer promise as substitutes for Co in WC-Co bodies.
3. Aluminum, silicon, manganese, and chromium are poor binders for wolfram carbide.
4. When iron, or nickel-chromium alloy were hot pressed with WC, a "case" was often formed. With iron, the case was slightly harder than the core; with nickel-chromium alloy, the case was slightly softer than the core. Possible applications of the case to ordnance problems should be investigated.
5. In hot pressing operations generally there occurred an extrusion of excess binder from the hot compact. Such extrusion of binder is probably desirable but the amount of excess binder and associated pressure, time, and temperature required to give a known final composition should be further explored.
6. Within the limits investigated, there proved to be little influence of WC grain size on the specific gravity of WC-Co compacts.
7. Indications are that with increasing grain size of WC in WC-Co compacts, the hardness decreases, and compression strengths decrease.

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I. INTRODUCTION

This project under the auspices of the Watertown Arsenal Laboratories was undertaken for two reasons, the first being to investigate substitute binders for cobalt in cemented wolfram carbide hardmetals and the second being to investigate the effects of grain size on certain physical properties of wolfram carbide cemented by cobalt.

Little work in the above fields has been systematically reported in the literature. The Germans (1) investigated the properties of carbides cemented by cobalt, nickel, iron and alloy binders made of each of the former paired with wolfram, molybdenum or chromium. Binders composed of nickel plus iron, iron plus nickel oxide, copper, silver oxide plus cobalt and others were investigated. Most of these "hardmetals" were tested by measuring the cutting life of a tool made of the composition being evaluated. Few actual hardness tests or compression tests were made.

Norton et al (2) further investigated some of the above binders and formulated recommendations on properties of binders considered for use with wolfram carbide to make "hardmetals" by the cold press-sinter techniques. The recommendations were that the binder should wet the tungsten carbide particles, not form a third phase, should be able to dissolve the carbide and should cause formation of a low melting liquid phase. In addition, it was found that nickel closely resembled cobalt and nearly satisfied the aforementioned requirements. It has been reported (3) that nickel, because it retains some of the carbide in solid solution, is inferior to cobalt. A portion of the present investigation was therefore concerned with the possibility of obtaining an alloy of nickel which could more closely approach the binder qualifications of pure cobalt. Lenel (4) reported that Fe and Ni were not good binders for WC compacts prepared by hot pressing using resistance sintering.

Since little other data is available on the properties of compacts prepared by hot pressing wolfram carbide with various binders, the hot pressing technique was considered to be worthy of investigation in the present work.

The second phase of this work, the relation between carbide grain size and the physical properties of wolfram carbide cemented by cobalt, has been noted in the literature (5,6). Recently Gurland and Bardzil (7) reported their results on the investigation of the relation between hardness, density, transverse rupture strength and grain size. They prepared the compacts by usual industrial methods and no special technique was used to influence the grain size. They found hardness and transverse rupture strength to vary

with grain size but the density showed no consistent variation. For the present work (undertaken before publication of the work of Gurland and Bardzil) the proposed technique was to start with a fine grained powder and by adjustment of time-temperature relationships, prepare sintered compacts having different average grain sizes.

Engle (8) has noted that the particle size of the milled powder determines the grain size of the sintered compacts. Grain growth has been reported at temperatures as low as 1250°C (below the Co-WC eutectic); however, the time necessary was 48 hours. Because of the accelerated growth at high temperatures (5), and the completeness of sintering after a short time at these temperatures (9), a temperature of 1500°-1500°C was chosen as the final sintering temperature for the second part of this project.

It was hoped that, by using hot pressing techniques, uniform, low porosity compacts could be prepared of varied grain sizes by either varying the hot pressing time or by re-sintering hot pressed compacts.

Hot pressing by resistance sintering has been used with some success to prepare sintered carbides. Cotter, Kohn, and Potter (10) found that the binder phase contained eta phase (Co_3W_3C) in large enough amounts to be found by X-ray techniques. This they attributed to the rapid quench associated with the technique used. Lenel (4) reported that with a high energy input coarse grains could be prepared but graphite would precipitate. Fine carbide grains were produced by using a low energy input; the grains thus produced were much finer than those produced by the conventional sintering techniques. This information indicated that the hot pressing method proposed above should be investigated although a different heating technique might be required.

Meerson and Shabalin (11) showed that if previously hot pressed compacts were heat treated, they increased in hardness. Such additional treatment was examined in the course of this work.

This project was closely coordinated with the Watertown Arsenal Laboratory and because of interest in iron as a substitute for cobalt (primarily because of iron's availability and cheapness) the greater part of the time allotted to this project was devoted to preparing and studying WC compacts cemented by iron.

II. EXPERIMENTAL TECHNIQUES

Hot pressing and cold pressing followed by sintering were used to make solid compacts from powdered constituents. The latter process was used for only the grain growth studies. The former was used for both grain growth and binder studies. Hot pressing was done by the common method of inductively heating a thermally insulated graphite die containing the powder (see Figure 1). Temperature was determined by a Leeds & Northrup optical pyrometer sighted on a 3/8 " deep 3/8 " diameter hole drilled in the die wall at the approximate position of the specimen. Temperature was manually held in a range of $\pm$ 15°C as closely as possible. Pressing was done on a 20 ton manual hydraulic press fitted with 3 gauges, two for the low pressures used in hot pressing and one for high pressures used in cold pressing. Cold pressed specimens were pressed in hardened steel dies. An oleic acid suspension of molybdenum sulfide* was applied to the steel die walls as a die lubricant. Prolonged sintering of "green" and hot pressed compacts was done in both a graphite crucible induction furnace and in a hydrogen furnace. Temperature was determined by either an optical pyrometer, thermocouple, or Rayotube.

The die designs used for hot pressing are shown in Figure 2. The die shown in Figure 2a produced a specimen 9/16 " diameter which was used for determination of density, hardness and microstructure. Figures 2b, and 2c show the designs of the double action dies used to prepare compression test specimens. The die in Figure 2a was loaded by placing 17 grams of powder in the chamber. The die in Figure 2b was loaded by making 4 to 5 green pressings of 5 to 8 grams each and loading these into the die until 30 grams of compacted powder was loaded. The die in Figure 2c was used to produce the cobalt bound compacts for grain size studies. This type of die was directly loaded with 30 grams of powder.

Powder mixtures were prepared by weighing out the powders in desired proportions, blending them with a spatula, mixing them in a one-quart neoprene lined ball mill with wolfram carbide balls and petroleum ether (boiling range 30°- 60°C). The mill was rotated at 80 R.P.M. After milling, the powder was dried and sifted through a 60 mesh sieve to remove chips from the wolfram carbide balls and to break up lumps of the powder. The powder was then ready for hot pressing. A lubricant, if used for cold pressing was added by making a thick slurry from the powder with a solution of the lubricant. This slurry was well mixed until the volatile solvent, usually benzene, had nearly evaporated. When almost dry the paste was spread on paper

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* Suggested by Mr. B. Bovarnick of Watertown Arsenal Rodman Laboratory.

towels until dry. It was then broken up with a spatula and bottled for use. The powder used to make the compacts used in the grain size studies did not contain an organic binder.

The following information was obtained about the metal powders used in this work:

Powder	Source	Size	Purity, %	Remarks
Al	Unknown	---	---	---
Co	Charles Hardy	0.7-1.0 micron	99.75-99.85	0.1-0.2% Ni
Cr	Charles Hardy	-300 mesh	99	Electrolytic
Cu	U.S. Metals Refining Co.	---	---	Type "c"
Fe	Antara Chemicals	---	---	Carbonyl, Type HP
Mn	Charles Hardy	-300 mesh	98-99	---
Ni	Charles Hardy	10-12 micron	99.9	Carbonyl
Si	Charles Hardy	-200 mesh	97 +	---
WC	Carboloy	1.5 micron ave. 6 micron max.	---	Total carbon 6.12 % Free carbon 0.05%

When testing alloy binders formed by mixing elemental powders it was hoped to achieve sufficiently intimate contact during ball milling so that rapid formation of low melting phases would occur.

Densities were determined by the displacement method by measuring the weight of the specimen in air, and submersed in a liquid of known specific gravity. Distilled water was used as the liquid except for the iron compression test specimens. For these carbon tetrachloride was used in order to reduce the possibility of rusting. Where specimens were too porous for this determination micrometer calipers were used to measure accurately the dimensions of the specimens and the volume was then calculated for use in density determinations.

Hardnesses were measured on a standard Rockwell Hardness tester using the "A" scale (60 Kg load).

Metallographic studies were made with a Zeiss Neophot large epi-microscope and camera. Specimens were prepared by cutting the compacts on a Di-Met diamond cutoff wheel. The sections were mounted in an air curing resin and polished in the following sequence.

1. Hand grind on glass plate with 400 mesh Norton boron carbide and water.
2. Hand grind using 600 mesh boron carbide and water.
3. Hand grind using 800 mesh boron carbide and water.

4. Polish on slow wheel using 1-5 micron Elgin Diamond Compound on AB microcloth.

5. Polish on slow wheel using 0-1/2 micron AB Di-Met Hyprez compound on AB microcloth.

The unetched polished specimens were examined and photographed at 200X for porosity determinations. The specimens were etched in the usual hot alkaline-ferricyanide solution* and were examined and photo[illegible]phed at 1500X. Where porosity photographs at 200X were inconclusive, unetched specimens were examined and photographed at 1000X. Grain size and porosity were estimated by using special charts, Figures 3, 4, 5 and 6, supplied by the Watertown Arsenal Laboratory.

Compression tests were carried out at Watertown Aresenal under the direction of Mr. Kenneth Abbott.

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* Solution composition: 10g $K_3Fe(CN)_6$, 10g KOH, 100 ml H_2O.

III. RESULTS AND DISCUSSION

A. SUBSTITUTE BINDER STUDIES

1. Iron

Iron was considered for this work because it has previously shown indication of being promising (2). The investigation on iron binders was expanded beyond original plans because of the great interest shown by Watertown Aresenal Laboratories in early very promising results obtained with iron. Iron binder compacts with specific gravities from 8.9 to 15.4, hardnesses from R_A48 to 94.1, and porosities from too large to measure to better than A-1* were prepared and studied. Several compression test specimens were prepared which had compression strengths of from 634,100 to 712,900 p.s.i. (Table 9).

One set of tests was made for effect of milling time on iron-wolfram carbide compacts. There did not seem to be much difference between one and five hours milling but the densities, hardnesses and porosities were adversely effected by a 24 hours milling. Accordingly, a 4 1/3 hour milling time was selected and used for most of the work with iron binders.

At 1200°-1225°C, 3500 p.s.i. there was an increase in both density and hardness as hold time at pressure increased (Table 1). The hardness values showed little increase in the range from 5 to 20 minutes dwell time. (The values for HP45Fe8 1/4 - 235 are included but are doubtful because of a possible mix-up which may have occurred between it and HP46Fe8 1/4 - 237). All densities of compacts formed at such low temperatures were less than theoretical.

Densities of compacts prepared at 1400°-1425°C increased as hold time lengthened from 1 to 10 minutes. Hardness values varied negligibly. All values for density were greater than theoretical indicating binder extrusion which probably continued, to a limit, as time lengthened.

There was no measurable change in grain size for pressings carried out at either 1200°or 1400°C. From subsequent experience with hot pressing of wolfram carbide-cobalt compacts, it appeared that even at high sintering temperatures grain growth during hot pressing was a relatively slow process and there was only slight growth after one to one and one half hours.

From Table 1 it may be seen that density increased with pressure at 1200°C. Hardness showed negligible change above 3500 p.s.i. However, the densities of compacts pressed at

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* ASTM tentative standard for porosity.

1200°C, 5200 p.s.i. for ten minutes were close to theoretical. For compacts pressed at 1400°C the densities increased slightly when pressing pressure increased from 3500 to 5200 p.s.i. but there was little hardness change. As would be expected, the porosity tended to decrease with increasing pressure.

Table 1, in which the 8 1/4% Fe-91 3/4% WC compacts are listed in order of increasing temperature, shows that a rapid compaction began around 1000°C. Compacts prepared by pressing at 1050° - 1075°C and 1100° - 1125°C at 3500 p.s.i. for 10 minutes were too porous to measure density by water displacement. Hardness and density values were lower than desired, and the specimens were not metallic in appearance. The specimens prepared at 1200°C and higher were metallic but densities near theoretical were not obtained at 1200°C until approximately 5200 p.s.i. pressure was held for 10 minutes. Specific gravities were 14.2-14.4 but porosity tended to be excessive. As the temperature of pressing was increased, density values of the compacts increased to greater than theoretical even at pressing pressures of 3500 p.s.i. At the highest pressing temperatures, 1400° to 1425°C, the specific gravity of the compacts was 15.3 and hardness was R_A93.8 for compacts which were hot pressed at 5200 p.s.i. for 10 minutes. A temperature increase may cause increased binder extrusion by creating greater plasticity and lengthening the time a plastic state exists. This is indicated by the higher hardnesses and greater specific gravities which characterized compacts pressed at 1400°C at lower pressure and for shorter dwell times.

Table 2 and Figure 7 show the effects of composition on the properties of the iron-wolfram carbide compacts. The compacts were prepared by hot pressing iron-wolfram carbide powders containing 2, 4, 6, 8 1/4, and 10% iron. The pressure, dwell time, amount of powder, and milling time were held constant at 3500 p.s.i., 10 minutes, 17 grams, and 4 1/3 hours respectively. For each composition, pressings were made at 1200°, 1300° and 1400°C. The data in Table 2 indicates that at 1200°C the specific gravity dropped steadily as the amount of iron increased from two to ten percent, and the values were always less than theoretical. The hardness of compacts prepared at 1200°C increased to a maximum of R_A93.2 for 8 1/4% iron and then dropped as the amount of iron was increased. The specific gravities of compacts prepared at 1300°C decreased until the iron content reached 6%. For compositions from 6% iron to 10% iron there was little change in the specific gravities of the compacts prepared at 1300°C. The experimentally determined specific gravity curve intersects the theoretical specific gravity curve at a point which corresponds to a composition of 4% iron and 96% wolfram carbide. The hardness of the compacts made at 1300°C is R_A93-93.6 for compositions

containing two to ten per cent iron. There was a slight increase in hardness as the iron content of the powder increased. The specific gravities of the compacts pressed at 1400°C decreased to a minimum of 15.0 for compacts made from powder containing larger amounts of iron, up to 10%. The curve of the experimentally determined specific gravities of iron-wolfram carbide compacts prepared at 1400°C lies above the theoretical specific gravity curve for all compositions containing more than 2.5% iron. There was a gradual, if really any, increase of hardness as the iron content of the powder increased.

A peculiar effect, shown in Figure 8 was observed in compression test compacts hot pressed from wolfram carbide powder containing 2% iron. Although the temperature and pressure were 1300°C and 4000 p.s.i., respectively, the powder apparently was unable to pack properly. For comparison, Figure 9 shows a properly hot pressed specimen prepared from a powder containing a higher percentage of iron.

The increasing specific gravities after a minimum was reached for compacts made from powder containing 6% iron probably resulted from greater binder extrusion. The reason for the 1400°C curve showing specific gravities nearly all greater than theoretical can be explained by the previously mentioned greater plasticity and lengthened time at temperature where the plastic state exists. The longer time at the plastic state resulted from the longer time required both to reach the dwell temperature and to cool down below the temperature range where plasticity was greatest. At most, the time differential was only six minutes. However, since the contents of the mold were plastic for a longer period, the binder, had a longer time in which to ooze to the surface thence out of the specimen. This would change the composition of the final specimen, hence the specific gravity and hardness values would actually be those for a compact having less binder. This would account for some compacts having specific gravities apparently greater then theoretical.

Mention should be made of the observation that the diamond "Brale" used to make the hardness determinations frequently caused the compact to fracture in the region surrounding the point of contact between the compact and the "Brale". However, hardness values did not appreciably differ from those where no fracture occurred.

The results of some compression tests of compacts of wolfram carbide bound by iron are listed in Table 9, together with the conditions under which the compacts were prepared. These compacts were pressed from wolfram carbide powder containing a nominal 10% iron. Some compacts were prepared from WC powder containing 2% iron but they were unsuitable for testing (see Figure 8). Although the compression test compacts were hot pressed like

the other smaller iron-wolfram carbide compacts in this work, it was necessary to use higher pressures on the larger dies in order to obtain compacts which had properties similar to those prepared on the small scale. This need for higher pressure probably resulted from the increased force lost in overcoming the greater die wall and interparticle friction associated with the die design and compact length needed for the compression test specimens. The compression strengths determined on these specimens ranged from 634,100 to 712,900 p.s.i. These values are in the same range as is covered by compacts of wolfram carbide bound with cobalt (8). There was no consistent change in compression strength that could be related to the change in hot pressing temperature from 1275° to 1400°C with pressure kept constant at 4000 p.s.i. There was also no variation that could be related to the change in hot pressing pressure of 4000 p.s.i. to 6100 p.s.i. while the temperature was kept constant at 1400°C.

The iron phase (Fe_3W_3C) equivalent to the eta phase, (Co_3W_3C) of the ternary system Co-W-C, was not reportedly found in compacts of iron and wolfram carbide containing less than 25% iron (12). In the present work, however, something like the eta phase of the Co-W-C system was occasionally observed metallographically but it may have been a result of the polishing technique used. No X-ray analysis was made to verify the presence of the eta-type phase.

There was but little evidence of grain growth; Figure 12 shows one of the few compacts where angular grains did grow. Specimens otherwise had an amorphous appearing structure in which often the powder particles seem to have coalesced (Figure 13). As may be seen from Table 1 and 2, the average grain size was in the range of 1-3 microns for the majority of the iron-wolfram carbide compacts. This was little change from the particle size of the starting powder.

The porosity ranged from higher than could be measured by the technique used, to better than A-1, as was mentioned previously. The porosity seemed most dependent on temperature, and for compacts pressed below 1200°C the porosity was usually excessive. For compacts prepared at approximately 1200°C the porosity became measureable and ranged from A-6 to A-3 depending somewhat on the amount of iron used and the time of dwell at pressure. When the compacts were pressed at 1300°or 1400°C the porosity was, with but a few exceptions, A-1. The variations were erratic but seemed to occur mostly for those compacts prepared from powder which was milled for 24 hours.

General conclusions from the work with iron binder are:

a. 4 1/3 hours milling time is sufficient for the techniques used.

b. Compacts having specific gravities greater than 13.6 and hardnesses greater than R_A85 can be prepared from wolfram carbide powder containing from two to ten nominal percent iron as a binder.

c. The binder may be extruded if the powder is pressed at high enough temperature.

d. Under certain conditions a case is formed (see section 8 below).

2. Nickel

For comparison purposes specimens of nickel-wolfram carbide were prepared by the hot pressing technique used in this work. Table 3 summarizes the data obtained. Here, as with iron, considerable binder extrusion was noticed. Specific gravities from 14.3 to 15.6, hardnesses from R_A80.0 to 93.0, compression strengths from 543,000 to 575,200 p.s.i., porosities from A-3 to A-1 and grain sizes from 2 to 3 micons up to 9 microns were obtained for specimens prepared in this manner from powder composed of 8 1/4% nickel and 91 3/4% wolfram carbide.

Little difference was apparent between properties of compacts pressed from powder composed of nickel and wolfram carbide which had been milled for 1 1/3 and four hours and pressed at 1400°C both at 3500 and 5200 p.s.i. When pressed to 3500 p.s.i. at 1300°C, wolfram carbide mixed with nickel powder and milled 1 1/3 hours gave higher density and hardness than powder milled for 24 hours, and even had possibly greater density than powder milled for 4 hours and pressed at a higher pressure of 5200 p.s.i. At the least, the nickel-wolfram carbide powders milled 1 1/3 hours and 4 hours, then hot pressed at 3500 and 5200 p.s.i., respectively, had nearly the same hardness and density values. Consequently, one to four hours milling time seemed adequate for the present work.

Nickel-wolfram carbide powder pressed to 3500 p.s.i. for one minute at a temperature of 1400°C gave compacts of slightly lower density than compacts prepared similarly but pressed for five and ten minutes. There was little difference in density and hardness values of the compacts prepared by pressing for five or ten minutes. All density values were greater than theoretical indicating extrusion of the binder. No data was obtained nor were compacts prepared in which the dwell time was varied at other temperatures.

Since there was negligible difference in density and hardness values for specimens prepared either at 1300° or at 1400°C from powder milled 1 1/3 and 4 hours, these values have been combined in Table 3 to show the effect of pressure and temperature.

The data in Table 3 show that when hot pressing powder composed of wolfram carbide and 8 1/4% nickel at 1200°C a pressure increase from 3500 to 5200 p.s.i. caused an eight point increase in hardness while there was little change in density. When the temperature was raised to 1300°and 1400°C a pressure increase caused little change in hardness and density.

Hardness of compacts tended to increase as the temperature at which they were pressed was increased. After the hardnesses reached 92.5 for compacts pressed at 1350°C there was little change in hardnesses for compacts prepared at higher temperatures. This may be seen from the hardness of 92.5 obtained for a compact pressed at 1500°C and at the higher pressure of 5200 p.s.i. Specific gravity values for the compacts of nickel-wolfram carbide were found to increase as the pressing temperature increased until a value of 15.3 was attained for compacts pressed near 1325°C. Compacts pressed at 1500°C showed only about 0.3 units gain in specific gravity.

Except for the effect of pressure on hardness of compacts prepared at 1200°C, the temperature seemed to have the strongest influence of all variables tested. The effect of temperature on the hot pressed compacts was perhaps most significant and critical between the 1300°-1325°C range and the 1325°-1350°C range where actual specific gravity values exceeded theoretical values. The latter effect may be, as noted previously, a result of extrusion of binder as evidenced by flashings which were found between plungers and the mold walls, and from the scarcity of binder observed metallographically.

Metallographic studies (Figure 14 shows typical microstructure) showed that the wolfram carbide grains tended to grow easily when nickel was used as a binder. The highest pressure and temperature used caused the greatest grain growth. The porosity was generally A-1 to A-3 for the hot pressed compacts. The high porosity at the highest specific gravities may have been a result of either the polishing technique or precipitation of graphite.

General conclusions concerning nickel binder are:

a. Milling time need be no longer than 4 hours.
b. Pressings can be made at 3500 p.s.i., 1300°-1325°C with 10 minutes hold at temperature. These pressings should retain most or all of the binder and should have low porosity and small grain size. Specific gravity is 14.6-14.8 and R_A hardness is 90-90.5.
c. Higher values of hardness and density can be attained at higher temperatures, from 1325°C up at least to 1500°C. These increases probably occur at the expense of binder loss. The

highest specific gravity and hardness values attained were in the ranges 15.3-15.6 and 90-93 R_A, respectively.

d. Either a higher temperature, longer dwell time, or both, somewhat compensate for lower pressure.

e. The critical binder extrusion temperature for 8 1/4% Nickel 91 3/4% WC starting composition is probably close to 1300°-1325°C where nearly theoretical density is obtained for powders milled for 1 1/3 hours, pressed at 3500 p.s.i., and milled powders pressed 4 hours at 5200 p.s.i.

3. Nickel Alloy Binders

a. Nickel-Chromium

The nickel-chromium, wolfram carbide mixture was chosen at 4.9% Ni, 5.1% Cr, and 90% WC because the nickel and chromium are in the proportions reported for the low melting (1343°C) eutectic of the binary system Ni-Cr (13).

Specific gravities from 13.9 to 14.6 and hardness values from R_A 85.5-92.3 (Table 4) were obtained for compacts prepared from the above powder. Porosity was low and average grain size was 1-2 microns. There was no evidence of a reaction between the WC and Cr of the alloy as was observed for the chromium, wolfram carbide hot pressed compacts. X-ray analysis might have shown the existence of possible reaction products but such work was not deemed essential at this time.

The specific gravities and the hardness values increased when the temperature of pressing was raised. The highest values, 14.5-14.6 and R_A 92.0-92.3 were recorded for specimens prepared from powder milled 1 1/6 hours and pressed at 5200 p.s.i., at 1500°-1530°C for 10 minutes. All of these compacts had a "case" which was softer than the center section by several R_A points (Table 7).

None of these compacts was fractured by the diamond "Brale" during a hardness test. This indicates a tougher compact than many of the others previously investigated. Here as with the pure nickel, cobalt and iron, there was evidence of the binder being extruded. Compacts of this composition are promising and should be further investigated.

The microstructure of these compacts is quite different from the usual cemented wolfram carbide "hardmetals" (Figure 15). In the unetched specimen what looked like pores at 200 X were actually long nearly white stringers of "lakes" of segregated binder when the unetched specimen was examined at 1000 X. Inside the larger of these areas were

smaller areas of a darker phase (Figure 16).* These phases have not been identified.

For hot pressed compacts of cemented wolfram carbide the nickel-chromium mixture shows promise as a binder; however, if the work of Norton et al (2) can be applied to hot pressed compacts, the existence of the third phase should be considered to be detrimental and undesirable.

The above mentioned "case" was found on all the wolfram carbide compacts cemented by the nickel-chromium mixture. The hardness increased from R_A 90.0-92.2 on the outer surfaces to R_A 93.8 in the center. The porosity of the case was less than the porosity of the center of a cross section, the plane of which was parallel to the direction in which the pressure was applied. Porosity at 200 X was difficult to determine because of the similarity between the segregated binder and the pores.

b. Nickel-Iron

There is no sharp, low melting eutectic reported for the binary system Fe-Ni (14), hence, a binder composed of 10% Fe and 90% Ni melting at approximately 1450°C was considered worthy of investigation. The resulting composition of the nickel-iron-wolfram carbide mixture was chosen as 9% Ni, 1% Fe, and 90% WC.

Specific gravities of the four compacts which were prepared were 14.6 to 15.5 while hardness values were R_A 90.0-93.7 (Table 4). From Table 4 it can be seen that, compared to the Ni-Cr-WC compacts, 10 minutes at a more moderate temperature range of 1300°-1325°C and at moderate pressure of 3500 p.s.i. was sufficient to produce densities close to theoretical. A rise of approximately 100°C caused the binder to be extruded as evidenced by the increase in hardness values, density values and the grain to grain contact observed in the microstructure.

The microstructure (Figures 17 and 18) of the wolfram carbide bound by the above compound binder is similar to that of some of the hot pressed nickel compacts having similar densities though slightly different starting compositions. Specimens of both types pressed at 1400°C and 3500 p.s.i. showed grain to grain contact while the specimens pressed at 1300°C showed lakes of segregated binder. The nickel-wolfram carbide compact pressed at 1300°C had a greater porosity than that of the nickel-iron-wolfram carbide compact even though the former was pressed at 5200 p.s.i. compared to 3500 p.s.i. for the latter.

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* It is interesting to note that the islands within the "lakes" also appeared on HP2Ni8 1/4-127.

c. Nickel-Copper

The composition, 1% Cu, 9% Ni, 90% WC was arbitrarily chosen because here again there is no low melting eutectic, high in nickel, in the binary system Ni-Cu (15). Since copper has been found to be a poor binder for wolfram carbide (2), it was maintained at only 10% of the total binder. Here again only four compacts were prepared and as may be seen in Table 4 the conditions of preparation were the same as for the iron-nickel compound binder tests. The resulting properties of the copper-nickel binder test compacts were strikingly similar to those of the iron-nickel binder compacts.

The similarity of the iron-nickel and the copper-nickel binder compacts was apparent also in the microstructure, Figures 19 and 20. Grain to grain contact appeared in the specimens pressed at 1400°C, while stringers of segregated binder appeared at 1300°C.

4. Manganese

Manganese, while not a member of the iron group of the periodic table, often resembles the members of this group in its properties. Manganese was accordingly tried as a binder for WC. The preliminary testing indicated that one hour of milling was best of the three milling times tried (Table 5).

Experiments showed that the required hardness and density could be obtained with an 8 1/4% Mn, 91 3/4% WC mixture. At 1300°C a pressure of about 5200 p.s.i. was applied to the die for 10 minutes and the resulting compacts had specific gravities of 13.8-13.9 and R_A hardness values of 91.5-92.1. The former were considerably below the calculated theoretical specific gravity of 14.5. From the high porosity (A-5), it was apparent that sintering was unsatisfactory. A typical microstructure of specimens of wolfram carbide-manganese compacts is shown in Figure 21. Experiments with lower percentages of Mn indicated that the porosity decreased and the density more closely approached theoretical density. For example, a 2% Mn, 98% WC compact had a specific gravity of 15.0, R_A hardness of 93.6-93.8 and an A-1 porosity when pressed 10 minutes at 1400°C at about 5200 p.s.i. Powder of the same composition pressed for 10 minutes at 1500°C and at the same pressure, even more closely approached theoretical specific density (15.1 vs. 15.2, theoretical) with very low porosity. Because of the porosity of the few specimens containing 8 1/4% Mn and the fact that the conditions for obtaining good compaction were above the melting point of Mn, work was discontinued on manganese and no compression tests were made.

The apparently poor binder properties of manganese even when hot pressed above its melting point and the resulting porosity (each pore may serve as center for stress concentrations) were reasons for considering manganese undesirable for a binder with wolfram carbide.

Perhaps with differently prepared manganese powder further work would prove more fruitful. However, even this may not be too practical since manganese itself is a critical strategic material.

5. Chromium

Because of its ability to dissolve carbon to a limited degree (16) and wolfram continuously (17) chromium was considered worth investigating as a substitute binder for cobalt in cemented wolfram carbide. Cemented carbide prepared from powder containing 8 1/4% Cr and 91 3/4% WC exhibited specific gravities from 12.4 to 14.0 with R_A hardness values from 62.7 to 94.2. Compression strength on a high density high hardness specimen was 489,100 p.s.i. (Table 9).

Apparently a reaction occurred during the hot pressing operation as evidenced by the low porosity (A-1) with a density below theoretical. The microstructure (Figure 22) was not the typical angular individual grains of WC surrounded by a matrix of binder. Instead the microstructure consisted of a series of interconnected areas of one phase containing small areas of another phase. Neither of the latter two phases was angular. From thermodynamical considerations it seems probably that a chromium carbide was formed.

Of the compacts made from powder milled 1, 4 and 24 hours, those made from powder milled 4 hours gave the highest values of specific gravity but the hardness values did not vary in a consistent manner. The compacts used in this comparison were prepared by hot pressing for 10 minutes at 5200 p.s.i. in the temperature range 1600°-1635°C. The hardness and specific gravity values of these compacts ranged from R_A 93.8-94.2 and 13.6-14.0, respectively (Table 6). The compacts prepared from the powder milled for four hours had specific gravities of 14.0 and R_A hardness values of 93.9-94.1.

Many of the compacts were fractured by the diamond "Brale" while measuring hardness (Figure 10). One broke while being removed from the mold. One of the compression test specimens broke while being ground. Because of this evidence of brittleness, the relatively low compression strength, and the high temperature needed to get high density, chromium did not appear to be a good binder for WC. The brittleness was probably caused

by the products of the previously noted reaction.

Two specimens were prepared for compression testing. Although nearly maximum available pressure was applied at 1600°C for 10 minutes the specific gravities of the large specimens were only 13.9 with R_A hardness values of 93.7-94.4. Both specimens were prepared from powder milled 4 hours. One of these specimens broke while being ground; the other specimen was tested and gave a compression strength of 489,100 p.s.i.

6. Silicon

Compacts prepared from powder containing 8 1/4% Si and 91 3/4% WC never sintered properly and no reliable density or hardness values could be obtained.

Powder of the above composition was milled for 1, 4 1/4 and 24 hours. Compacts from the resulting powders were made at 1350°-1400°C, 5200 p.s.i. for 10 minutes, but all compacts though slightly metallic were undersintered and extremely porous. Since the compacts were so poor even though prepared at a temperature close to the reported melting point of silicon it was decided not to pursue this investigation any further. The possibility of a chemical reaction being the basic cause of poor compact formation was considered but not proved.

7. Aluminum

One specimen of wolfram carbide with aluminum as a binder was prepared. The powder composition was 0.5 weight per cent aluminum, 99.5% wolfram carbide. The powder was milled one hour. While hot pressing, the powder showed no inclination to compact suddenly, a property observed in all the hot pressing where the binder produced a metallic appearing compact. Although the temperature was raised to 1000°C before the test was discontinued, this specimen, like the specimens tested with silicon as a binder, was extremely undersintered. The melting point of aluminum is 660°C so a noticeable compaction would be certainly expected before 1000°C was reached. The powder was of uncertain nature and age. The well known thin film of aluminum oxide which rapidly forms on aluminum surfaces probably contributed heavily to the behavior of the powder; nevertheless, it seems probable that the film would rupture under pressure if the inner metal of a particle became molten. There is a possibility that by use of hydrides this trouble could be eliminated (18). As an example, powdered lithium aluminum hydride could be used and the lithium distilled off after the hydrides had decomposed.

8. The "Case" Effect

An interesting result of the hot pressing of both iron-wolfram carbide and chromium-nickel wolfram carbide compacts was the frequent formation of a "case". This case was apparent on polished specimens when examined with the unaided eye. It is referred to as a "case" because of its resemblance to the case which is produced in case hardened steel. A typical polished cross section exhibiting a case is shown in Figures 11, 23 and 24.

All of the chromium-nickel-wolfram carbide compacts which were prepared for this work also displayed the case; however, the case pattern was not always the same for every cross section. The hardness of the centers of the cross sections of the chromium-nickel-wolfram carbide compacts was greater than that of the edges (Table 7). The porosity of the edges was less than in the center of the cross sections.

Many of the compacts made from iron-wolfram carbide also showed a case as noted in Tables 1 and 2. From these tables it appears that the formation of a case was somehow related to the amount of binder and the temperature. For compacts prepared from wolfram carbide powder containing 2% iron there was a case formed on compacts pressed at 1200°C, but no case for compacts pressed at 1300° and 1400°C. A case appeared for compacts hot pressed at 1400°C from wolfram carbide powder containing 4, 6 and 8 1/4% iron. All compacts pressed from powder containing 10% iron had a "case". The hardness of the centers of the cross sections of the iron-wolfram carbide compacts was less than that of the edges. The relation between temperature, per cent binder and the appearance of the case in the compacts indicate that the case is related to binder extrusion.

Other possible causes of the case effect are:

a. The innermost particles mechanically jamb together but not before enough binder has been squeezed to the edges to lubricate the outer particles and enable them to rearrange and compact better.
b. Surface carburization.
c. The binder is first squeezed out of the edges, because of higher pressures at the surfaces, and forms a case which seals in the inner binder.
d. Other chemical reaction at the surface (e.g. b).

Suggestion "a" can be eliminated because the porosity of the edges in the iron compacts was greater than the center section; also there appeared metallographically to be more binder in the center section than in the edges.

Suggestions "b" and "d" are quite possible but need further investigation.

Suggestion "c", in view of the present evidence, seems quite plausible. If a seal were formed at the edges, the binder would be present in larger amounts in the interior of the compact; this is found to be true. The formation of a case in the compacts prepared from powder of higher iron content may be explained by extusion of the binder at the edges; when the binder at the edges is gone the grains are so close that there are very few channels through which the interior binder can reach the surface. Thus the binder in the center is trapped and forms lakes while at the surface there is no excess binder except for a thin film of binder so grain to grain contact occurs. In the compacts prepared with 2% iron there was no excess binder; throughout the entire specimen grain to grain contact occurred almost immediately when pressure and temperature were high enough to cause compaction. The case which appeared at 1200°C for the compacts made from 2% iron was not apparent in compacts made from the same powder pressed at the same pressure at 1300° and 1400°C. At 1300° and 1400°C the powder charge was more plastic hence, pressure was more evenly distributed throughout the compacts.

The phenomena of a case resulting from carburization has been mentioned by the German investigators (19). Their report does not clearly describe the case, hence it is difficult to compare the formation with that observed in the present work. They described the case in connection with nickel-wolfram-graphite powder which was pressure sintered. The case was attributed to carburization of the wolfram.

B. GRAIN GROWTH STUDIES

The study of various methods of producing wolfram carbide-cobalt compacts having different average grain sizes was a preliminary step considered necessary to the study of the effect of grain growth on the physical properties of the compacts. The methods considered for making compacts with different grain sizes from the same starting powder mixture were:

1. Hot press for varying hold times.

Hot press for a short time at low temperatures and pressures until barely sintered. Resinter in a furnace.

3. Hot press for a short time at high temperatures. Resinter in a furnace.

4. Cold press. Sinter for varying lengths of time.

The above techniques were considered with the view that one or more could be used to prepare dense compacts of low porosity and controlled grain size variation.

Attempts to prepare grain growth study specimens by hot pressing for different lengths of time were rather unsuccessful; however, no hot pressing was made for more than 1.5 hours at 1550°C and at 1000 p.s.i. Moreover, eta phase frequently appeared in such hot pressings.

It was hoped that hot pressing followed by resintering would be a good technique because prolonged hot pressing found necessary to grow grains, is time consuming and might introduce excessive graphite. Also it was hoped that the desirable properties of the hot pressed compacts, especially low porosity, might be retained after the sintering. However, these hot pressed compacts generally tended to swell and exhibit enlarged pores during the resintering operation. This occurred with or without use of an organic binder-lubricant in the starting powder. The enlarged pores were concentrated in the inner region of the compacts and were thought to be caused by expansion of trapped gases at higher temperatures. It has been suggested that hydrogen can diffuse into a compact and react under sintering conditions to form molecules too large to escape (20). This action was thought to continue until the built up pressure is relieved by expansion of the pore. During the present work, enlarged pores have been observed in compacts which were sintered in hydrogen and non-hydrogen containing atmospheres. Since neither hydogen nor organic binder were present when the pore enlargement occurred, the conclusion was reached that the enlargement was due to gases sealed in the compact during the initial hot pressing. This suggested that if specimens were hot pressed at or above the resintering temperature, the enlarged pores resulting from resintering could be eliminated. Likewise, vacuum hot pressing might offer promise. Unfortunately time limitations prevented the testing of these possibilities. A few specimens treated by the former suggestion showed little porosity increase, even though grain growth occurred, after one hour and two hours resintering. Another method of avoiding enlarged pores which could be used would be to hot press at 1100° or 1200°C for a short time at low pressure. The barely sintered compact which resulted could then be refired at a higher sintering temperature. Since there was little difference between this product and a presintered cold pressed compact, it seemed reasonable to expect good compacts from this technique. This technique did produce some compacts having A-1 porosity but also a few with high porosity.

Since the previously discussed hot pressing and hot pressing followed by resintering were only moderately successful in preparing specimens for grain size studies, cold pressing was finally selected for the present work. For the technique used the lowest porosity was

obtained for compacts prepared from a powder containing no organic binder. This eliminated the time consuming "baking out" of the organic binder. Comparisons showed little difference between compacts sintered in either a hydrogen furnace or a graphite induction furnace once the atmosphere of the latter was adjusted to prevent formation of eta phase and graphite in the sintered compacts.

Norton, Gurland and Rautala (21), and Dawihl (22) mentioned the critical adjustments of sintering atmosphere with respect to the appearance of eta or graphite in compacts of wolfram carbide cemented by cobalt. This was also found to be true in the preliminary work on grain growth which preceded the studies on effects of grain size. In preparing the cobalt-wolfram carbide compacts by cold pressing then sintering many of the compacts showed the converted eta phase referred to by the Germans (23). The converted eta phase occurred in hot pressed specimens which were further sintered in a hydrogen furnace while packed in alumina in a graphite boat. Eta also occurred in hot and cold pressed specimens fired in a graphite induction furnace with or without dry hydrogen atmosphere. This phase was also found to occur in a specimen sintered in a graphite induction furnace while another specimen sintered simultaneously, but packed in a small graphite crucible filled with graphite dust and placed adjacent to the unpacked specimen, showed no eta phase but contained precipitated graphite. This is similar to the observation of the Germans (24); hence, as they suggested, a packing of graphite mixed with fused alumina was used to prevent eta and graphite formation. Eta phase and graphite were finally eliminated by using alumina which had been mixed with graphite and then freed of most of the graphite by pouring it from container to container before an electric fan. The resulting alumina contained a small amount of graphite which was enough to prevent eta phase without causing excessive graphite precipitation in the sintered compacts. Pure alumina was not tried and by itself may prevent occurrence of the eta phase or graphite.

The compacts for use in the study of the effects of grain size on properties were finally prepared by cold pressing 30 grams of the powder to 36,000 p.s.i. in a 3/8" diameter lubricated steel die. These green compacts were sintered in a graphite induction furnace. The specimans were packed in alumina containing a trace of graphite dust. The temperature for the sintering and grain growth was maintained as close to $1520^{\circ} \pm 6^{\circ}C$ as the technique permitted. The times for sintering at this temperature were 1, 2 and 4 hours. These firing times produced compacts having a good range of grain sizes.

The grains did not grow uniformly and the microstructure usually consisted of large

grains surrounded by many much smaller grains, Figures 26, 27 and 28. Because of the non-uniformity of grain sizes, the grain size data in Table 8 indicates the average sizes of both the small and large grains which compose the compact. Uniform grain growth was not expected because no special blending of powder sizes was used to promote uniform grain growth; instead, a fine powder was used which had an average particle size of 1.5 microns with a maximum particle size of 6 microns.

The conditions of preparation and the resulting properties of the compacts finally used for studying effects of grain size are listed in Table 8.

From consideration of the data shown in Table 8 there seems to be little influence of wolfram carbide grain size on density. Any difference was so slight as to have little significance in view of the small number of compacts which were studied and may even be due to porosity differences as suggested by the porosity data. When considering hardness, however, there was a marked decrease in average hardness values as the grain sizes of the compacts increased. The hardness values given in Table 8 are the average values for the two ends of the sintered compact. From these data it is apparent that generally the low values (in the left hand hardness column of Table 8) for compacts of a given grain size were still higher than the high values for the group of compacts having the next larger grain size. This trend corresponds with previously reported relations between grain size and hardness and density data (5,6,7). From Table 8 it can be seen that, in general, development of large grains causes a significant lowering of the compression strength. It would be worth further investigation to confirm this result on compacts prepared, if possible, with a more uniform size of grain in the various ranges of grain size. The data obtained in this work confirm the opinions and data obtained at high strain rates that large grains have an undesirable effect on the strength.

Some specimens which were hot pressed from the same powder as used above were included for comparison. These were compacts for which the density was close to those of the cold pressed sintered compacts. The hot pressing technique, however, enables production of the completely sintered compact before the grains could grow very large (Figure 25). Although there was a difference in densities between these hot pressed compacts and the cold pressed sintered compacts there was probably no difference in composition because no binder extrusion was observed. The hardness values of the hot pressed compacts were higher and the grain size, 1-2 microns, was smaller than cold pressed compacts which were sintered for one hour; they had a grain size of 2-3, + 7 microns, and were the hardest of the specimens prepared by the cold press-sinter technique.

The density of the hot pressed specimens was higher than any of the cold pressed specimens prepared in this work and is accounted for by the low porosity. From experience with binders other then cobalt, it has been apparent that for hot pressing techniques, hardness and density increase at different rates with respect to each other. Hardness may reach a value after which it changes little while specific gravity continues to increase. Because of this fact the hardness of the hot pressed specimens can be considered with some reliability to be the hardness of specimens having densities equivalent to that obtained by cold pressing followed by sintering. Hence, it seems reasonable at least to consider the relation between grain size and hardness to be the same for the hot pressed as for the cold pressed and sintered compacts.

The compression strengths of two hot pressed compacts were 617,000 and 697,500. These compacts were uniformally fine grained. The average grain size was 1-2 microns. These compression strengths are in a range similar to that covered by the cold pressed specimens having the smallest grain size.

In all of the above work, note should be made of the relatively small compression test specimens used: nominally 3/4" long X 3/8" diameter. The extreme difficulty of eliminating any possibility of bending and/or eccentric loading would ordinarily require tests be done on more specimens. However, the data for Table 9 are certainly indicative of the influence of grain size on strength in compression.

C. GENERAL REMARKS

Of the many possible binders tested as substitutes for cobalt in cemented wolfram carbide, iron was outstanding; hot pressed test specimens made with iron binder had high density, great hardness, and low cost (as compared to the cost of cobalt-containing compositions). Further and more comprehensive work on the iron-wolfram carbide system is definitely recommended.

Aluminum, and silicon appear to be especially poor substitutes for cobalt; hard dense compacts could not be prepared using these metals as binders. Manganese showed some promise but evidently did not wet or dissolve the wolfram carbide to the degree desired; consequently, the compacts tended to be porous until relatively high temperatures were used. Chromium mixed with wolfram carbide produced good, hard, fairly dense compacts; however, the chromium apparently reacted with the wolfram carbide and the compacts were brittle. Alloy binders of nickel and chromium, nickel and iron, and nickel and copper

were quite promising, the latter two especially being worthy of further investigation. The hardness of wolfram carbide compacts cemented by the nickel-copper alloy was higher than the hardness of compacts pressed from wolfram carbide containing only nickel. This resulted although the alloy binder-wolfram carbide compacts were made at lower pressure and temperature than the nickel-wolfram carbide compacts. In addition, the wolfram carbide compacts made with the alloy binder were less porous and had a slightly larger grain size than the compacts bound with nickel. The nickel-iron alloy binder produced compacts which, all controllable variables being constant, were remarkably similar to the compacts using the nickel-copper alloy as a binder. The differences in hardness and densities were negligible for compacts prepared in the same way from wolfram carbide powder containing 9% nickel plus either 1% iron or 1% copper. The only difference was that at 1400^{o}C the wolfram carbide grains in the compacts cemented by the copper-nickel alloy were larger than those in the similarly prepared compacts using the iron-nickel alloy.

Compacts made using nickel-chromium alloy were neither as hard nor as dense as those prepared at lower temperature and pressure using copper-nickel or iron-nickel alloys. Also all of the few compacts made from the nickel-chromium-wolfram carbide powder had a case which was softer than the core of the compact.

The most significant conclusion drawn from this work is that iron binder is potentially able to replace cobalt binder for certain ordnance objectives. Iron appears so promising from several standpoints that much more research and development work should be done, and larger shapes made and tested in actual use. Alloy binders of nickel plus iron or copper also offer interesting possibilities. Aluminum, silicon, manganese, and chromium are poor binders for wolfram carbide.

The conclusions drawn from the work on grain size are that the hardness decreases, specific gravity is little effected and compression strength decreases with an increase in grain size.

Suggestions for further work in addition to those made throughout this report are:

1. A comprehensive study should be made on iron as a binder.
2. Binders should be investigated which are powders of alloys, instead of elemental powders mixed in the proportions that exist in alloys.
3. Other nickel alloys should be studied as possible binders for wolfram carbide.
4. Comparisons should be made between hot pressed compacts were no binder is extruded and compacts from which binder has been extruded, but which have the

same final composition. Partial extrusion of binder should be studied as a means of achieving very low porosities.

5. Vacuum hot pressing should be investigated.
6. The "case" effect should be studied further to determine its potential applications to ordnance problems.
7. Heat treatable steels should be investigated as possible binders for WC.

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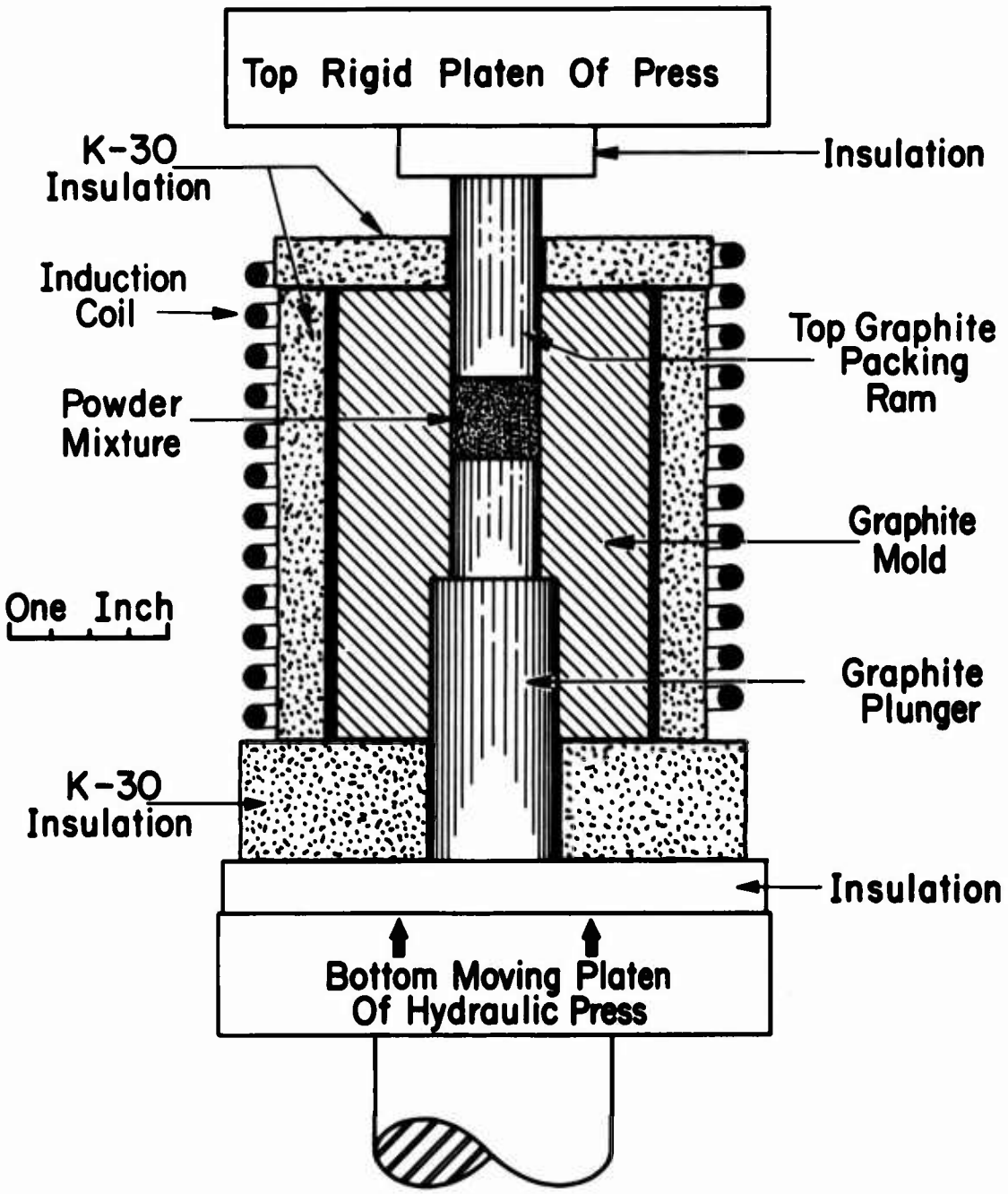
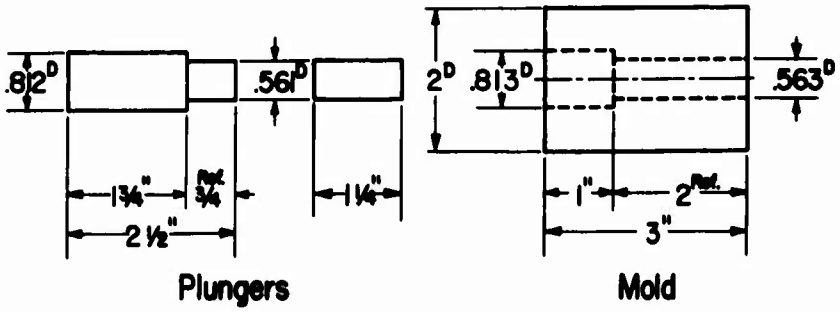
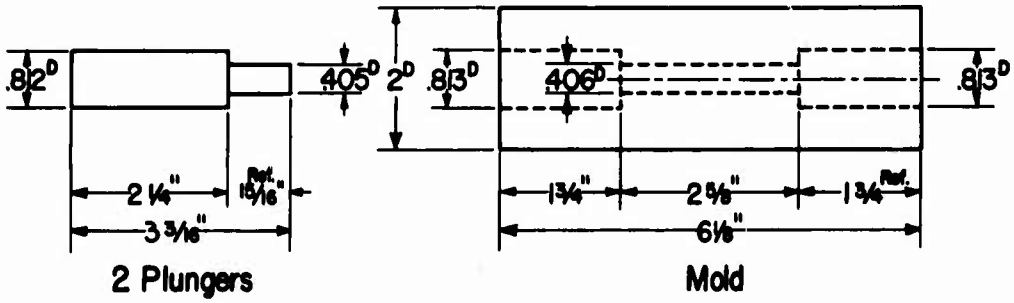


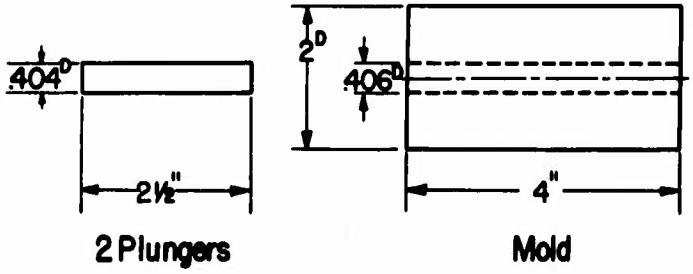
Figure 1. Hot Pressing Set-up.



2a. Substitute Binder Specimen Die



2b. Compression Test Specimen Die



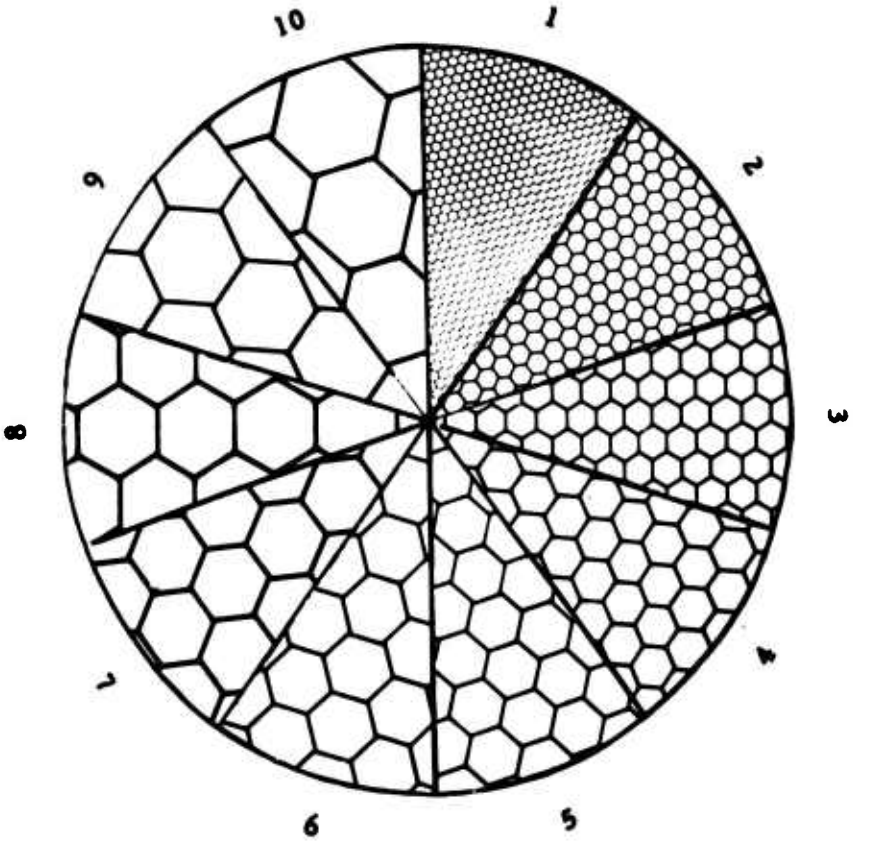
2c. Grain Size Test Specimen Die

Figure 2. Three Die Designs Used in Hot Pressing

CARBIDE GRAIN SIZE CHART

1 to 10 Microns at 1500X

1 Micron = .00003937 in.



WTN.639-9435

Figure 3. Grain Size Chart

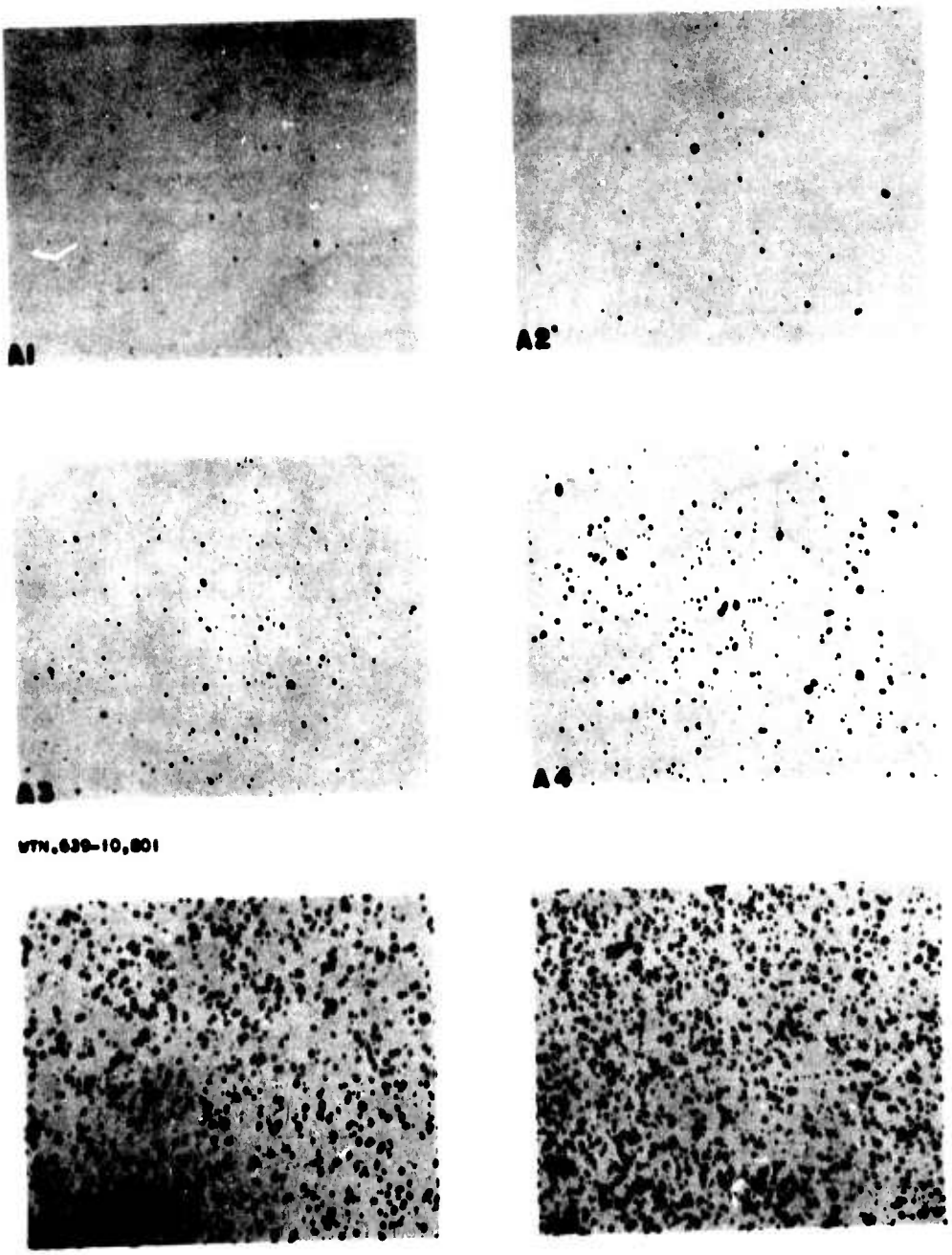


Figure 4. Porosity Chart A

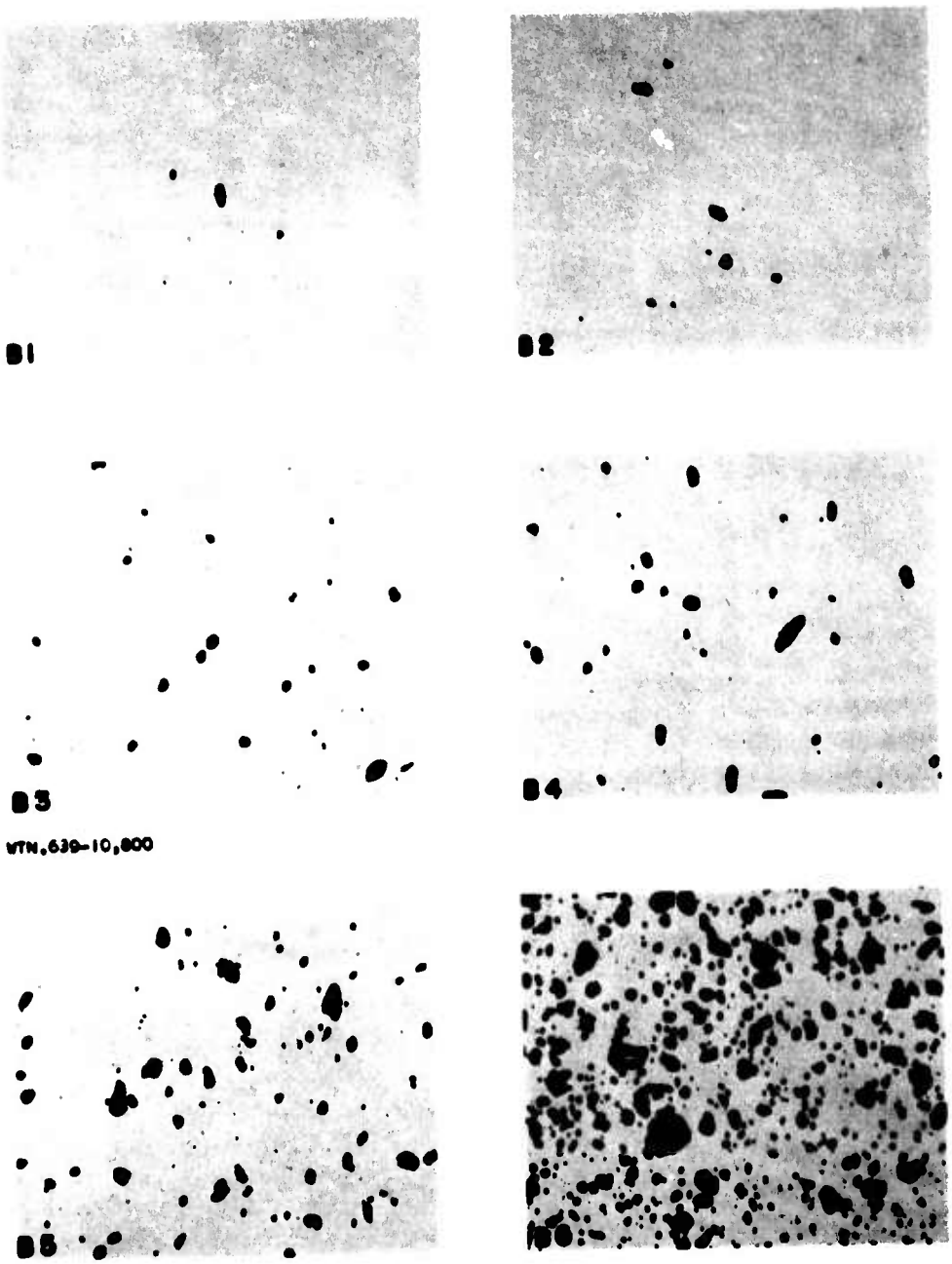


Figure 5. Porosity Chart B

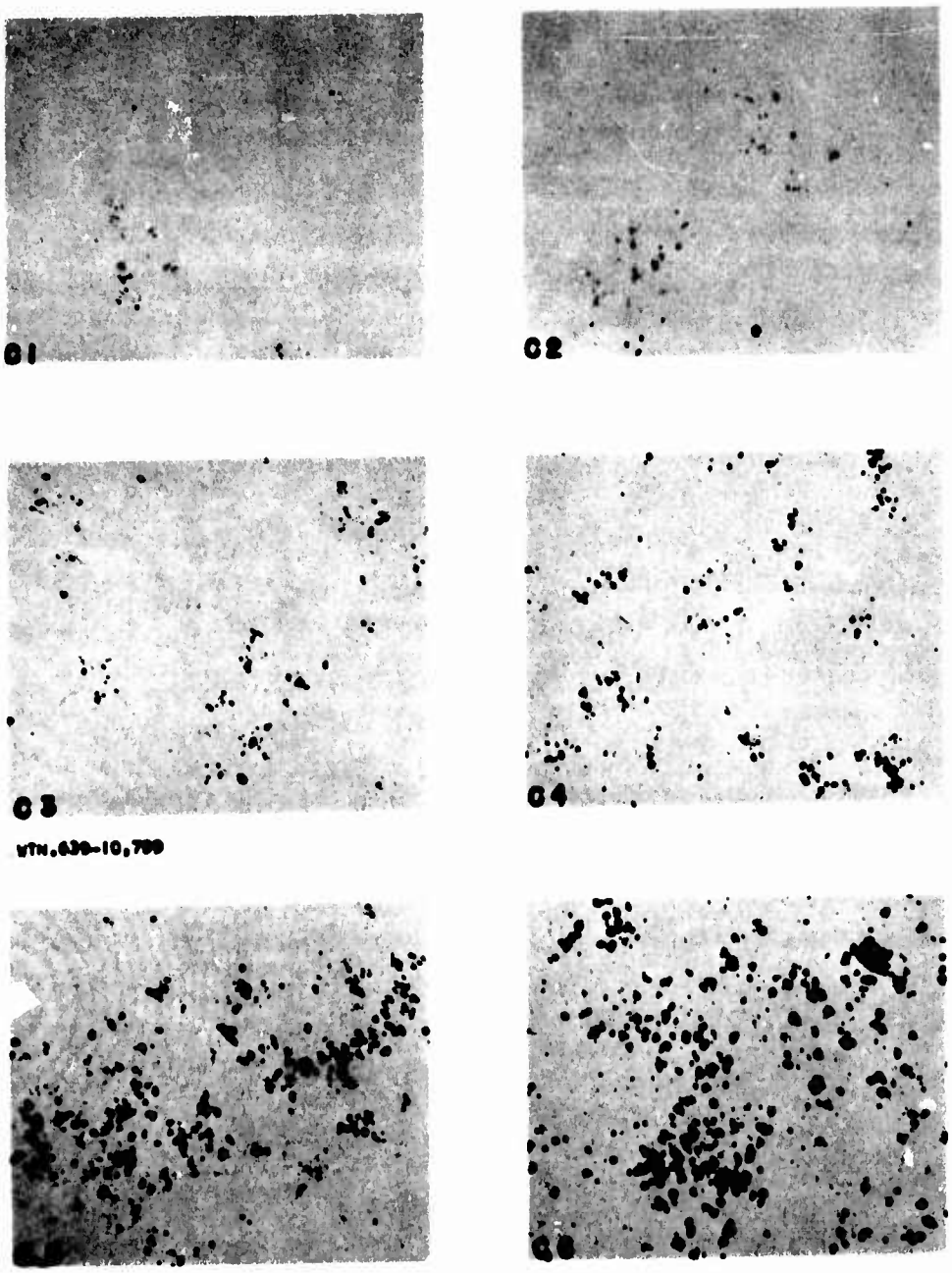


Figure 6. Porosity Chart C

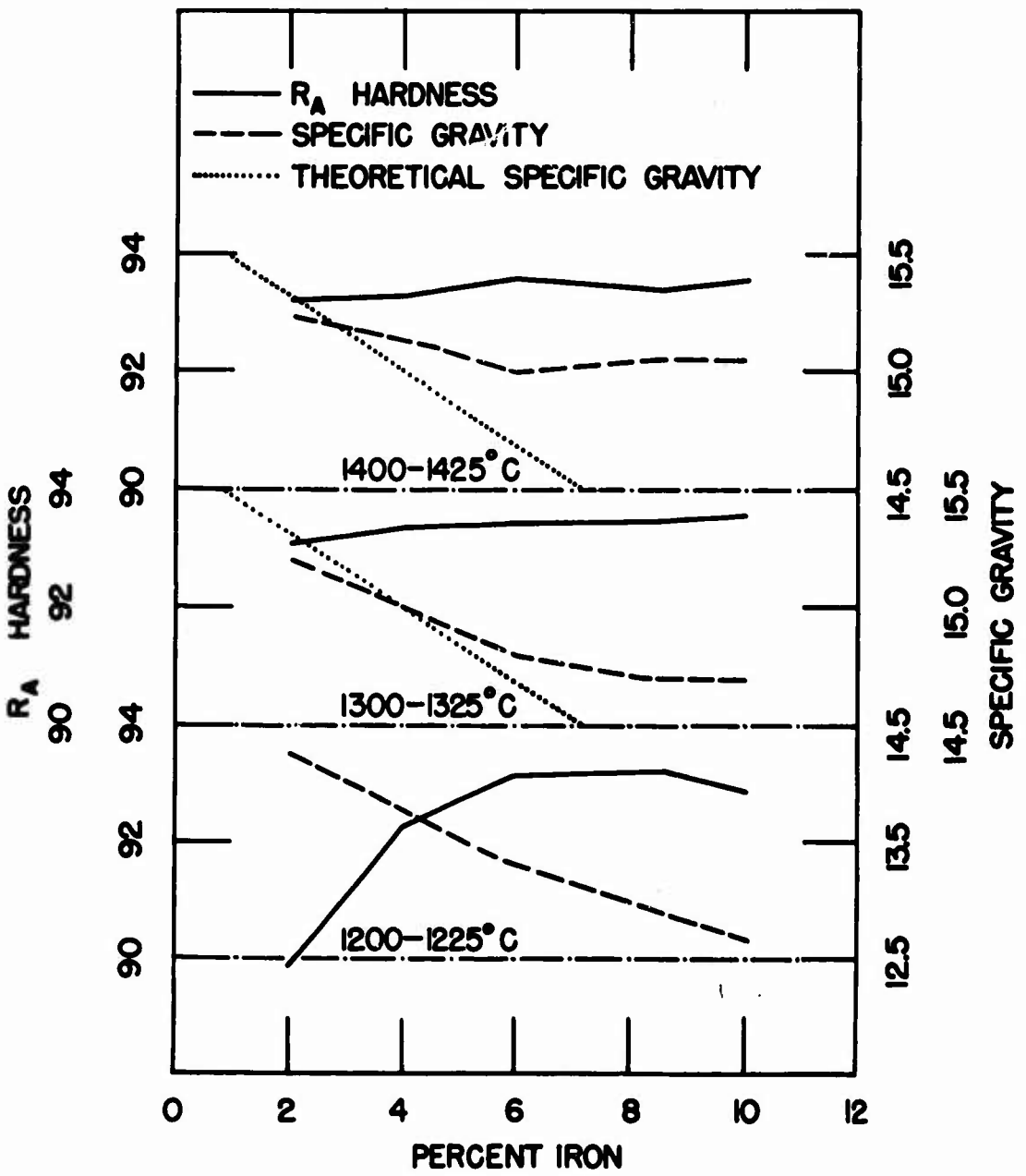


Figure 7. Graph Showing Change in Specific Gravity and in R_A Values with Change in Percentage of Fe at Various Temperatures at 3500 psi Pressure

Figure 8
Compression Test Specimen 2% Fe 98% WC
Poorly Sintered

Figure 9
Compression Test Specimen 10% Fe 90% WC
Properly Sintered

Figure 10
Specimen Showing Fractures produced by "Brale" of Hardness Tester

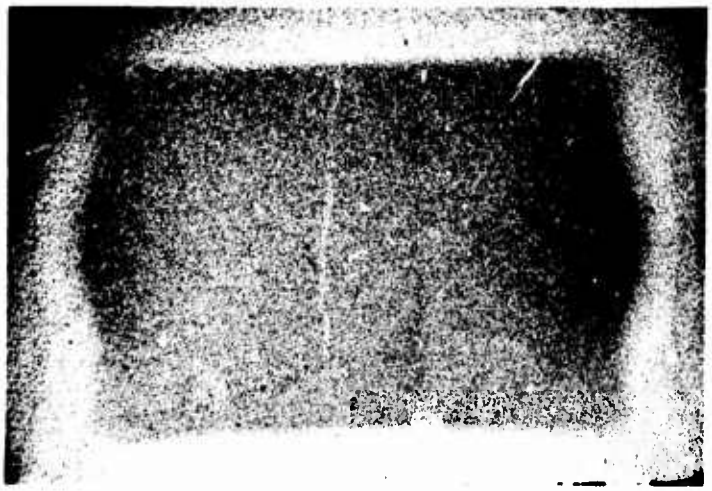


Figure 11
Typical Case 8X Etched

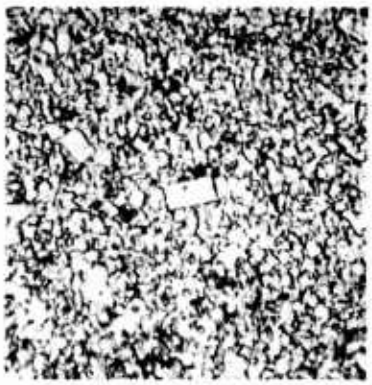


Figure 12
8 1/4% Fe 91 3/4% WC
Showing Larger Grains
1500 X Etched

Figure 13
4% Fe 96% WC Typical
Microstructure
1500 X Etched

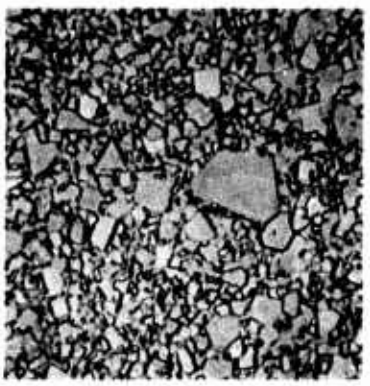


Figure 14
8 1/4% Ni 91 3/4% WC
Typical Microstructure
1500 X Etched

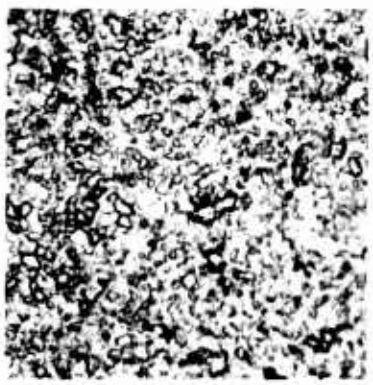


Figure 15
9% Ni 1% Cr 90% WC
Typical Microstructure
1500 X Etched

Figure 16
9% Ni 1% Cr 90% WC
200 X Unetched

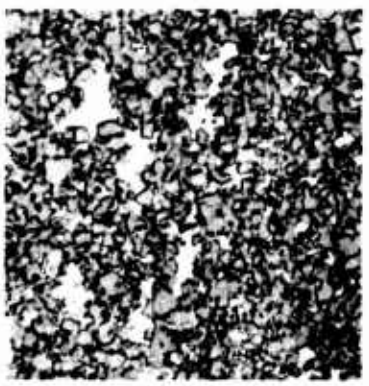


Figure 17
9% Ni 1% Fe 90% WC
1500 X Etched

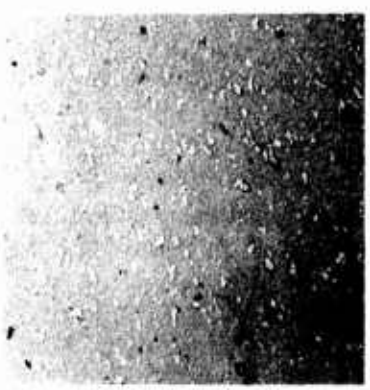


Figure 18
9% Ni 1% Fe 90% WC
200 X Unetched

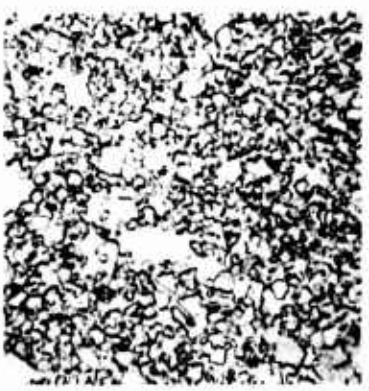


Figure 19
9% Ni 1% Cu 90% WC
1500 X Etched

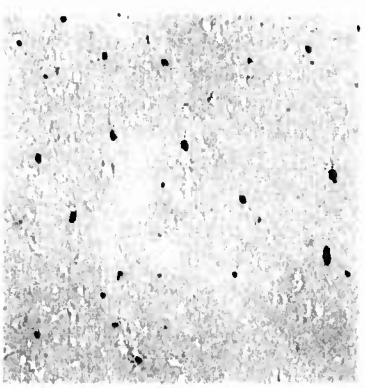


Figure 20
9% Ni 1% Cu 90% WC
200 X Unetched

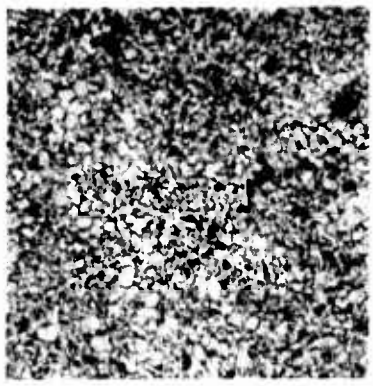


Figure 21
4% Mn 96% WC
1500 X Etched

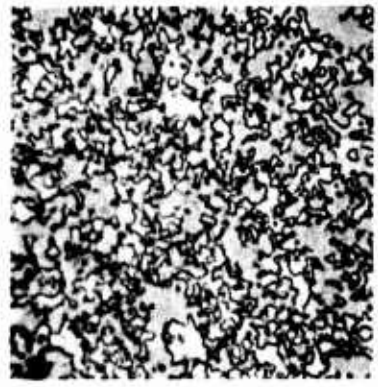


Figure 22
8 1/4% Cr 91 3/4% WC
1500 X Etched

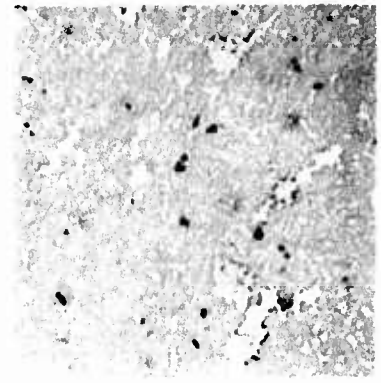


Figure 23
10% Fe 90% WC Center of Cross Section through Case 1500 X Unetched

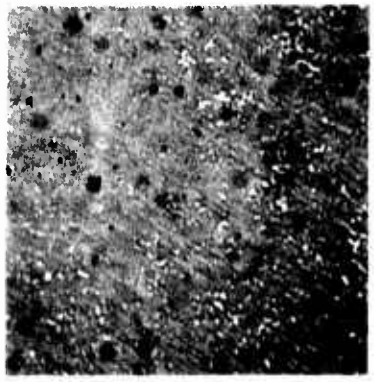


Figure 24
10% Fe 90% WC Edge of Cross Section through Case
1500 X Unetched

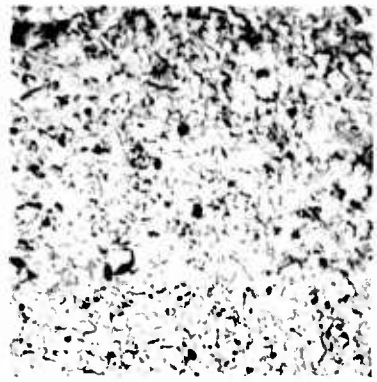


Figure 25
10% Co 90% WC Fine Grained, Hot Pressed
1500 X Etched

Figure 26
10% Co 90% WC Cold Pressed, Sintered 1 hour
1500 X Etched

Figure 27
10% Co 90% WC Cold Pressed, Sintered 2 hours
1500 X Etched

Figure 28
10% Co 90% WC Cold Pressed, Sintered 4 hours
1500 X Etched

TABLE I

Hardnesses, Specific Gravities and Conditions of Preparation of

8 1/4% Fe, 91 3/4% WC Binder Test Compacts

Specimen	Hours Milled	Sp. Gr.	R_A high	R_A low	Time at Pressure (mins.)	Temperature °C	psi Pressure	Possible Extrusion	Fracture by Brale	Porosity	Micron Grain Size	Comment
HP20Fe8 1/4 183	4 1/3	8.9	51.5	46.7	10	1055 - 1077	3500	N	N[1]	too spongy	too spongy	too porous
HP21Fe8 1/4 185	4 1/3	8.5	48	-	10	1050 - 1075	3500	N	N	"	"	" "
HP22Fe8 1/4 187	4 1/3	9.6	68.0	67.3	10	1103 - 1125	3500	N	N	"	"	" "
HP23Fe8 1/4 189	4 1/3	9.5	66.3	64.1	10	1100 - 1125	3500	N	N	"	"	" "
HP30Fe8 1/4 203	4 1/3	10.5	71.9	71.5	10	1103 - 1119	5200	N	N	"	"	" "
HP31Fe8 1/4 205	4 1/3	10.4	72.3	71.6	10	1102 - 1127	5200	N	N	"	"	" "
HP7Fe8 1/4 55	24	13.4	91.2	90.4	10	1140 - 1168	5200	N	N	A-3	1-2	" "
HP9Fe8 1/4 71	24	13.7	91.9	91.8	10	1145 - 1180	5200	N	Y[2]	A-6	-	Case (3)
HP46Fe8 1/4 237	4 1/3	13.5	93.2	92.9	10	1201 - 1223	1900	N	Y	A-6	1-2	Case
HP47Fe8 1/4 239	4 1/3	11.4	91.9	90.3	10	1193 - 1222	1900	N	Y	A-6+	?	"
HP42Fe8 1/4 229	4 1/3	10	84.0	82.3	1	1198 - 1220	3500	N	Y	A-6+	?	"
HP43Fe8 1/4 231	4 1/3	10	83.5	76.6	1	1201 - 1224	3500	N	N	A-6+	?	"
HP40Fe8 1/4 225	4 1/3	12.3	93.0	92.9	5	1205 - 1222	3500	N	Y	A-6+	?	"
HP41Fe8 1/4 227	4 1/3	12.2	92.9	92.9	5	1198 - 1222	3500	N	Y	A-6+	1-2	"
HP24Fe8 1/4 191	4 1/3	13.4	93.3	92.9	10	1198 - 1225	3500	N	Y	A-4	1-2	Case, binder segregation
HP25Fe8 1/4 193	4 1/3	13.2	93.3	93.2	10	1200 - 1222	3500	N	Y	A-3	1-2	" " "
HP38Fe8 1/4 221	4 1/3	13.1	93.3	93.2	10	1200 - 1230	3500	N	Y	A-5	1-2	"
HP39Fe8 1/4 223	4 1/3	13.1	93.2	93.0	10	1200 - 1230	3500	N	Y	A-5	1-2	"
HP44Fe8 1/4 233	4 1/3	14.0	93.1	93.1	20	1195 - 1228	3500	N	Y	A-3	1-2	Case, binder segregation in stringer
HP45Fe8 1/4 235	4 1/3	11.4	91.2	90.8	20	1197 - 1227	3500	N	Y	A-6	? ?	Case, maybe actually HP46
HP32Fe8 1/4 207	4 1/3	14.2	93.4	93.3	10	1200 - 1228	5200	N	Y	A-4	1-2	"
HP33Fe8 1/4 211	4 1/3	14.3	93.6	93.4	10	1198 - 1221	5200	N	Y	A-5	1-2	"
HP34Fe8 1/4 213	4 1/3	14.3	93.5	93.3	10	1195 - 1226	5200	N	Y	A-2	1-2	Case, binder in "lakes"

TABLE I (Continued)

Specimen	Hours Milled	Sp. Gr.	R_A high	R_A low	Time at Pressure (mins.)	Temperature °C	psi Pressure	Possible Extrusion	Fracture by Brale	Porosity	Micron Grain Size	Comment
HP35Fe8 1/4 215	4 1/3	14.4	93.7	93.6	10	1201 - 1226	5200	N	Y	B-1	1-2,+4	Case, binder in "lakes"
HP26Fe8 1/4 195	4 1/3	14.7	93.5	93.4	10	1302 - 1326	5200	Y	Y	A-1+	1-2	Grain to grain contact, fine binder in "lakes"
HP27Fe8 1/4 197	4 1/3	14.7	93.5	93.2	10	1303 - 1323	3500	Y	Y	A-1+	1-2	Slight case, grain to grain contact, fine binder "lakes"
HP36Fe8 1/4 217	4 1/3	15.0	93.9	93.8	10	1301 - 1325	5200	N	Y	A-1+	1-2	Slight case
HP37Fe8 1/4 219	4 1/3	14.9	94.0	94.1	10	1303 - 1324	5200	Y	Y	A-2	1-2	No case
HP28Fe8 1/4 199	4 1/3	15.0	93.4	93.4	10	1401 - 1427	3500	Y	Y	A-1	1-2	No case, grain to grain contact
HP29Fe8 1/4 201	4 1/3	15.1	93.5	93.2	10	1402 - 1423	3500	Y	Y	A-1+	1-2	
HP8Fe8 1/4 57	24	14.6	92.5	92.2	1	1400 - 1420	5200	Y	Y	A-3	1-2, +3	
HP10Fe8 1/4 73	24	14.5	92.5	92.2	1	1405 - 1407	5200	Y	Y	A-1	1-2	
HP11Fe8 1/4 75	24	14.8	92.9	92.7	5	1400 - 1425	5200	Y	Y	A-4	1-2, +4	
HP12Fe8 1/4 77	24	14.8	92.9	92.5	5	1390 - 1420	5200	Y	Y	A-4	1-2	
HP1Fe8 1/4 27	1	15.1	93.8	93.2	10	1405 - 1435	5200	N	Y	A-1	1-2	
HP3Fe8 1/4 35	1	15.4	93.8	93.7	9	1417 - 1430	5200	N	Y	A-3	1-2	
HP13Fe8 1/4 79	1	15.3	93.8	93.7	10	1400 - 1430	5200	Y	Y	A-1	1-2	Case
HP17Fe8 1/4 87	1	15.2	93.9	93.6	10	1407 - 1425	5200	N	Y	A-1	-	Slight case, non-metallic inclusions in unetched specimens
HP15Fe8 1/4 83	4	15.3	93.8	93.8	10	1405 - 1425	5200	N	Y	A-1	1-2	Case?
HP16Fe8 1/4 85	4	15.2	93.7	93.5	10	1410 - 1425	5200	Y	Y	A-1	1-2	"
HP2Fe8 1/4 31	5	15.3	93.4	93.3	10	1395 - 1430	5200	Y	Y	A-1	3-4	
HP4Fe8 1/4 37	5	15.4	93.5	93.3	10	1385 - 1440	5200	Y	Y	A-2	-	
HP6Fe8 1/4 45	24	14.9	92.4	92.2	10	1400 - 1425	5200	N	Y	?	2-3, +5	
HP18Fe8 1/4 93	24	15.0	93.2	93.1	10	1403 - 1425	5200	N	Y	-	-	
HP19Fe8 1/4 95	24	15.0	93.4	93.1	10	1398 - 1425	5200	Y	Y	-	-	
HP5Fe8 1/4 43	24	15.0	92.2	92.0	10	1395 - 1430	5200	Y	Y	A-3	1-2	

(1) N = no
(2) Y = yes
(3) Case = See Section IIIA

TABLE II

Effects of Composition on Hardness and Specific Gravity of Fe-WC Compacts Pressed at 1200°C, 1300°C and 1400°C

Specimen	Hours Milled	Sp. Gr.	R_A high	R_A low	Time at Pressure (mins.)	Temperature °C	psi Pressure	Binder Extrusion	Fracture by Brale	Porosity	Micron Grain Size	Comment
HP1Fe2 - 243	4 1/3	14.2	90.2	89.7	10	1202 - 1235	3500	N[1]	Y[2]	A-3	1-2	Case
HP2Fe2 - 245	4 1/3	14.3	89.8	89.7	10	1198 - 1221	3500	N	Y	A-3	1-2	"
HP3Fe2 - 247	4 1/3	14.3	89.9	89.8	10	1200 - 1235	3500	N	Y	A-3	1-2	"
HP4Fe2 - 249	4 1/3	15.2	93.1	93.0	10	1298 - 1318	3500	N	Y	A-1	2-3	Very slight case on 2 corners some grains angular
HP5Fe2 - 251	4 1/3	15.2	93.1	93.1	10	1280 - 1332	3500	N	Y	A-2	2-3	No case, some grains angular
HP6Fe2 - 253	4 1/3	15.2	93.4	93.0	10	1402 - 1423	3500	N	Y	C-1	2-3	No case, slight angularity
HP7Fe2 - 255	4 1/3	15.3	93.3	93.2	10	1403 - 1425	3500	N	Y	A-1	2-3	" " " "
HP1Fe4 - 257	4 1/3	13.6	92.2	92.2	10	1201 - 1227	3500	N	Y	A-3	1-2	Case, binder segregation
HP2Fe4 - 259	4 1/3	13.9	92.3	92.1	10	1202 - 1226	3500	N	Y	A-3	1-2	Case
HP3Fe4 - 261	4 1/3	15.0	93.4	93.2	10	1300 - 1325	3500	N	Y	A-1	2-3	Slight case
HP4Fe4 - 263	4 1/3	15.0	93.5	93.3	10	1285 - 1335	3500	N	Y	A-1	1-2 + 3	Case
HP5Fe4 - 265	4 1/3	15.2	93.2	93.2	10	1406 - 1422	3500	N	Y	A-1	-	No case
HP6Fe4 - 267	4 1/3	15.1	93.5	93.3	10	1402 - 1424	3500	N	Y	A-1	1-2	" "
HP1Fe6 - 269	4 1/3	13.5	93.1	93.0	10	1200 - 1226	3500	N	Y	A-6+	-	High porosity case
HP2Fe6 - 271	4 1/3	13.1	93.1	93.1	10	1202 - 1223	3500	N	Y	A-6+	-	High porosity, double case
HP3Fe6 - 273	4 1/3	14.8	93.5	93.4	10	1300 - 1325	3500	N	Y	A-1	1-2	Case
HP4Fe6 - 275	4 1/3	14.8	93.5	93.3	10	1306 - 1323	3500	N	Y	A-3	1-2 + 4	"
HP5Fe6 - 277	4 1/3	15.0	93.5	93.4	10	1398 - 1424	3500	Y	Y	A-1	2-3	No case
HP6Fe6 - 279	4 1/3	15.0	93.7	93.6	10	1395 - 1425	3500	N	Y	A-1	1-2 + 4	" "
HP1Fe10- 281	4 1/3	12.7	93.1	92.9	10	1198 - 1227	3500	N	Y	A-6 +	-	Double case
HP2Fe10- 283	4 1/3	12.6	93.1	92.4	10	1204 - 1230	3500	N	Y	A-6 +	-	Case
HP3Fe10- 285	4 1/3	14.7	93.6	93.3	10	1295 - 1337	3500	Y	Y	A-1	2-3	"
HP4Fe10- 287	4 1/3	14.7	93.7	93.5	10	1297 - 1328	3500	Y	Y	A-1	1-2	"
HP5Fe10- 289	4 1/3	15.0	93.6	93.5	10	1407 - 1427	3500	Y	Y	A-1	2-3	Slight case

TABLE II (Continued)

Specimen	Hours Milled	Sp. Gr.	R_A high	R_A low	Time at Pressure (mins.)	Temperature °C	psi Pressure	Binder Extrusion	Fracture by Brale	Porosity	Micron Grain Size	Comment
HP6Fe10- 291	4 1/3	15.1	93.6	93.3	10	1403 - 1424	3500	Y	Y	A-1	1-2	Slight case
HP7Fe10- 327	4 1/3	14.2	93.5	93.0	10	1250 - 1269	3500	-	-	A-2	-	Case
HP8Fe10- 329	4 1/3	14.4	93.3	93.1	10	1254 - 1273	3500	-	-	A-1	-	"
HP9Fe10- 331	4 1/3	14.5	93.5	93.5	10	1275 - 1297	3500	-	-	A-1	-	"
HP10Fe10-333	4 1/3	14.6	93.3	93.6	10	1275 - 1304	3500	-	-	A-1	-	"

(1) N = no
(2) Y = yes

TABLE III

Hardnesses, Specific Gravities and Conditions of Preparation of 8 1/4%Ni, 91 3/4% WC Binder Test Compacts

Specimen	Hours Milled	Sp. Gr.	R_A high	R_A low	Time at Pressure (mins.)	Temperature °C	psi Pressure	Possible Binder Extrusion	Fracture by Brale	Porosity	Micron Grain Size	Comment
HP12Ni8 1/4 - 147	1 1/3	14.3	80.8	80.0	10	1200 - 1225	3500	N(1)	N	-	-	
HP13Ni8 1/4 - 149	1 1/3	14.4	80.3	79.4	10	1200 - 1225	3500	N	N	-	-	
HP16Ni8 1/4 - 155	1 1/3	14.3	87.9	88.0	10	1200 - 1225	5200	N	N	-	-	
HP17Ni8 1/4 - 157	1 1/3	14.3	88.2	88.0	10	1200 - 1225	5200	N	N	-	-	
HP14Ni8 1/4 - 151	1 1/3	14.7	90.4	90.3	10	1300 - 1325	3500	Y(2)	N	-	-	
HP15Ni8 1/4 - 153	1 1/3	14.8	90.3	90.3	10	1300 - 1325	3500	Y	N	-	-	
HP25Ni8 1/4 - 173	24	14.4	88.5	88.0	10	1300 - 1325	3500	N	N	-	-	
HP26Ni8 1/4 - 175	24	14.6	89.3	88.9	10	1300 - 1325	3500	Y	N	-	-	
HP1Ni8 1/4 - 125	4	14.7	90.5	90.0	10	1285 - 1327	5200	Y	N	A-1	2-3	Binder in lakes
HP2Ni8 1/4 - 127	4	14.6	90.4	90.1	10	1300 - 1330	3500	Y	N	A-1	2-3	Binder in small lakes with islands in the lakes
HP27Ni8 1/4 - 179	4	15.3	90.1	90.0	10	1325 - 1350	3500	Y	N	-	-	
HP28Ni8 1/4 - 181	4	15.3	89.8	89.4	10	1325 - 1350	3500	Y	Y	-	-	
HP23Ni8 1/4 - 169	4	15.4	92.5	92.4	10	1350 - 1375	3500	Y	Y	A-1	2-3,+ 6	Slight grain growth
HP24Ni8 1/4 - 171	4	15.4	92.9	92.8	10	1350 - 1375	3500	Y	Y	A-1	2-3,+ 5	" " "
HP18Ni8 1/4 - 159	1 1/3	15.4	92.9	92.9	10	1400 - 1425	3500	Y	N	A-1	2-3,+ 5	Possible "hot spot"
HP10Ni8 1/4 - 143	1 1/3	15.5	92.4	92.3	10	1400 - 1425	3500	Y	Y	A-1	2-3,+ 4	Slight grain growth
HP9Ni8 1/4 - 141	1 1/3	15.6	93.0	92.8	10	1403 - 1425	5200	Y	Y	A-1	1-2,+ 6	Fair grain growth
HP8Ni8 1/4 - 139	4	15.5	92.7	92.6	10	1404 - 1465	3500	Y	Y	A-1	2-3	Slight grain growth
HP3Ni8 1/4 - 129	4	15.5	93.0	92.8	10	1396 - 1422	5200	Y	Y	A-4 ?	4-5 ?	" " "
HP4Ni8 1/4 - 131	4	15.5	92.6	92.5	10	1396 - 1427	5200	Y	Y	A-1	2-3	Very slight grain growth
HP6Ni8 1/4 - 135	4	15.5	92.9	92.8	10	1405 - 1424	5200	Y	Y	A-3	2-3,+ 5	Slight grain growth
HP5Ni8 1/4 - 133	4	15.6	92.5	92.5	10	1505 - 1530	5200	Y	Y	A-2	2-3,+ 7	Considerable grain growth
HP7Ni8 1/4 - 137	4	15.6	92.8	92.6	10	1497 - 1525	5200	Y	Y	A-3	2-3,+ 9	" " "
HP19Ni8 1/4 - 161	1 1/3	15.2	92.4	92.3	1	1403 - 1417	3500	Y	Y	A-3	1-2	Grain to grain contact with rounded edges on WC
HP20Ni8 1/4 - 163	1 1/3	15.3	92.8	92.7	1	1404 - 1407	3500	Y	Y	A-3	1-2	" " " "

(1) N = no
(2) Y = yes

TABLE IV

Hardnesses, Specific Gravities, Compositions and Conditions of Preparation of WC Compact Bound by Nickel Alloys

Specimen	Hours Milled	Sp. Gr.	R_A high	R_A low	Time at Pressure (mins.)	Temperature °C	psi Pressure	Binder Extrusion	Fracture by Brale	Porosity	Micron Grain Size	Starting Composition	Comment
HP4 $\frac{Ni\ 49}{Cr51}$ 10-341	1 1/6	13.9	86.0	85.5	10	1298 - 1332	3500	N[1]	N	A-1	1-2	4.9%Ni 5.1%Cr 90% WC	Soft cases visible Cr carbide possible, "Ponds" in lakes of binder Also crack near inside case
HP3 $\frac{Ni\ 49}{Cr51}$ 10-339	1 1/6	14.2	90.8	90.1	10	1302 - 1324	5200	N	N	A-2	1-2	"	
HP5 $\frac{Ni\ 49}{Cr51}$ 10-343	1 1/6	14.1	90.3	90.1	10	1290 - 1333	5200	-	N	A-1	Continuous	"	Evidence of additional phase after etching; stringers of binder visible in unetched specimen; case stringers + lakes of binder surrounding grey inclusion case visible
HP6 $\frac{Ni\ 49}{Cr51}$ 10-345	1 1/6	14.2	90.0	90.6	10	1350 - 1374	5200	Y[2]	N	A-1	Skeleton	"	
HP7 $\frac{Ni\ 49}{Cr51}$ 10-347	1 1/6	14.4	91.2	91.2	10	1360 - 1373	5200	-	N	A-1	"	"	
HP1 $\frac{Ni\ 49}{Cr51}$ 10-335	1 1/6	14.5	92.0	91.9	10	1500 - 1530	5200	Y	N	A-1	"	"	
HP2 $\frac{Ni\ 49}{Cr51}$ 10-337	1 1/6	14.6	92.3	92.2	10	1500 - 1526	5200	Y	N	A-1	"	"	
HP3 $\frac{Ni\ 90}{Fe10}$ 10-407	1	14.7	90.1	90.0	10	1275 - 1335	3500	N	N	A-1	1-2	9%Ni 1%Fe 90%WC	Lakes of binder
HP4 $\frac{Ni\ 90}{Fe10}$ 10-409	1	14.6	90.2	90.2	10	1300 - 1330	3500	N	N	A-2	1-2	"	" " "
HP1 $\frac{Ni\ 90}{Fe10}$ 10-395	1	15.5	93.5	93.5	10	1387 - 1425	3500	Y	Y	A-3	2-3	"	Grain to grain contact
HP2 $\frac{Ni\ 90}{Fe10}$ 10-397	1	15.5	93.7	93.4	10	1405 - 1430	3500	Y	Y	A-1	2-3	"	" "
HP3 $\frac{Ni\ 90}{Cu10}$ 10-411	1	14.6	90.0	90.0	10	1300 - 1320	3500	N	N	A-2	2-3+4	9%Ni 1%Cu 90%WC	Lakes of binder Slight grain growth
HP4 $\frac{Ni\ 90}{Cu10}$ 10-413	1	14.6	90.0	89.9	10	1300 - 1322	3500	N	N	A-2	2-3	"	-
HP1 $\frac{Ni\ 90}{Cu10}$ 10-401	1	15.5	93.4	93.3	10	1402 - 1425	3500	N	Y	A-1	3-4	"	-
HP2 $\frac{Ni\ 90}{Cu10}$ 10-403	1	15.5	93.4	93.3	10	1400 - 1426	3500	N	Y	A-3	3-4	"	-

(1) N = no
(2) Y = yes

TABLE V

Hardnesses, Specific Gravities, Compositions and Conditions of Preparation of Mn, WC Binder Test Compacts

Specimen	Hours Milled	Sp. Gr.	R_A high	R_A low	Time at Pressure (mins.)	Temperature °C	psi Pressure	Binder Extrusion	Fracture by Brale	Porosity	Micron Grain Size	Starting Composition	Comment
HP1Mn8 1/4- 29	1	13.9	92.1	91.5	10	1300 - 1315	5200	N(1)	Y(2)	A-5	2-3	8 1/4% Mn, 91 3/4% WC	
HP2Mn8 1/4- 33	1	13.8	91.9	91.6	10	1295 - 1330	5200	N	Y	-	-	"	
HP3Mn8 1/4- 39	4	13.6	91.9	91.5	10	1280 - 1320	5200	N	Y	-	-	"	
HP4Mn8 1/4- 41	4	13.7	91.3	91.1	10	1295 - 1317	5200	N	Y	-	-	"	
HP5Mn8 1/4- 47	24	13.2	90.1	90.1	10	1306 - 1320	5200	N	Y	A-5	1-2	"	
HP6Mn8 1/4- 49	24	13.5	90.5	90.4	10	1297 - 1317	5200	N	Y	-	-	"	
HP7Mn8 1/4- 67	24	13.6	91.2	91.1	10	1385 - 1450	5200	Y	Y	A-4	1	"	Porosity at 200 X appears to be inclusions & binder at 1000 X, 3rd phase appears on etching
HP8Mn8 1/4- 69	24	13.6	91.3	90.8	10	1412 - 1430	5200	N	Y	A-6	1-2	"	
HP1Mn2 -297	1	11.1	79.3	77.6	10	1206 - 1226	5200	N	N	-	-	2% Mn, 98% WC	
HP2Mn2 -299	1	11.1	77.5	76.8	10	1190 - 1235	5200	N	N	-	-	"	
HP3Mn2 -301	1	14.2	90.9	89.6	10	1299 - 1320	5200	N	Y	-	-	"	
HP4Mn2 -303	1	14.0	90.5	89.5	10.5	1280 - 1324	5200	N	Y	-	-	"	
HP5Mn2 -305	1	14.9	92.9	92.8	10	1395 - 1424	5200	N	Y	A-2	1-2	"	Porosity at 200 X appears to be binder & inclusions at 1000 X, 3rd phase appears on etching. Light grey phase (binder) at 1000 X unetched looks like pores at 200 X, 3rd phase appears on etching
HP6Mn2 -307	1	15.0	93.1	93.0	10	1402 - 1435	5200	N	Y	A-1	1-2	"	
HP7Mn2 -309	1	15.1	93.8	93.6	10	1500 - 1521	5200	N	Y	A-1	1-2	"	
HP8Mn2 -311	1	15.1	93.8	93.5	10	1507 - 1527	5200	N	Y	A-1	1	"	
HP1Mn4 -315	1	-	78.8	77.9	10	1202 - 1223	5200	N	N	-	-	4% Mn, 96% WC	Too spongy for determination
HP2Mn4 -317	1	-	77.1	75.0	10	1197 - 1225	5200	N	N	-	-	"	"
HP3Mn4 -319	1	14.1	-	-	10	1285 - 1335	5200	N	Y	-	-	"	
HP4Mn4 -321	1	14.1	91.1	90.3	10	1301 - 1321	5200	N	Y	-	-	"	
HP5Mn4 -323	1	14.8	92.5	92.2	10	1394 - 1435	5200	N	Y	A-1	1	"	Same as 305
HP6Mn4 -325	1	14.7	92.9	92.6	10	1402 - 1427	5200	N	Y	A-2	1-2	"	"

(1) N = no
(2) Y = yes

TABLE VI

Hardnesses, Specific Gravities and Conditions of Preparation of
8 1/4% Cr, 91 3/4% WC Binder Test Compacts

Specimen	Hours Milled	Sp. Gr.	R_A high	R_A low	Time at Pressure (mins.)	Temperature °C	psi Pressure	Binder Extrusion	Fracture by Brale	Porosity	Micron Grain Size	Comments
HP1Cr8 1/4- 59	1	12.4	69.5	62.7	10	1417 - 1445	5200	N(1)	N	-	-	
HP2Cr8 1/4- 61	1	13.3	84.9	83.4	10	1505 - 1530	5200	N	N	-	-	
HP3Cr8 1/4- 63	1	13.5	92.7	90.4	10	1585 - 1630	5200	N	N	-	-	
HP4Cr8 1/4- 65	1	13.4	92.4	91.4	10	1590 - 1630	5200	N	N	-	-	
HP5Cr8 1/4- 89	4	14.0	94.0	93.9	10	1605 - 1625	5200	N	Y(2)	A-1	Continuous	No angular WC grains, more like a continuous interconnected skeleton, possible Chromium reacted.
HP6Cr8 1/4- 91	4	14.0	94.1	94.0	10	1603 - 1630	5200	N	Y	A-2	"	Quasi - continuous structure
HP7Cr8 1/4- 99	24	13.6	94.2	94.0	10	1600 - 1635	5200	N	N	A-2	"	"
HP8Cr8 1/4-101	24	13.6	94.2	94.2	10	1600 - 1635	5200	N	Y	A-1 +	"	"
HP9Cr8 1/4-103	24	13.7	94.1	94.1	10	1712 - 1730	5200	N	Y	A-1	"	"
HP10Cr8 1/4-105	24	13.6	93.9	93.8	10	1700 - 1725	5200	N	Y	A-1	"	"

(1) N = no
(2) Y = yes

TABLE VII

Hardnesses on Ends and on Cross Sections of Compacts* Having "Cases"

	Rockwell "A" on Center of Cross-Section	Rockwell "A" on Ends of Cross-Section	Hardness Change Ends to Center
HP1Fe4 -257	86.8	92.2	decrease
HP35Fe8 1/4-215	91	93.6	decrease
HP2Fe4 -259	89.5	92.2	decrease
HP9Fe8 1/4 - 71	92	91.8	no change
HP9Fe10 -331	91.1	93.5	decrease
HP8Fe10 -329	90.4	93.2	decrease
HP7Fe10 -327	89.4	93.0	decrease
HP34Fe8 1/4-213	90.2	93.4	decrease
HP3Fe8 1/4 - 35	93.8	93.7	no change
HP40Fe8 1/4-225	77	93	decrease
HP42Fe8 1/4-229	71.0	81.5	decrease
HP43Fe8 1/4-231	68.9	72.0	decrease
HP44Fe8 1/4-233	86.3	92.7	decrease
HP45Fe8 1/4-235	62.8	90.3	decrease
HP47Fe8 1/4-239	68.8	89.8	decrease
HP1Fe6 -269	84.7	92.8	decrease
HP1Fe10 -281	92.3	78.1	decrease
HP2$\frac{Ni49}{Cr51}$10 -337	93.8	92.2	increase
HP1$\frac{Ni49}{Cr51}$10 -335	93.6	92.0	increase
HP3$\frac{Ni49}{Cr51}$10 -339	93.1	90.1	increase
** HP5Fe10 -289	93.4	93.5	no change
** HP6Fe6 -279	93.6	93.7	no change
** HP5Fe6 -277	93.8	93.7	no change

* Not all compacts with cases were tested.

** Compacts having no "case" included to show hardness of center and ends of cross-section are the same.

TABLE VIII

Variation of Physical Properties with Grain Size for 10% Co-90% WC Compacts Cold Pressed to 18 tsi then Sintered for 1, 2, and 4 hours at 1520 ± 6°C

Specimen No.	Approx. Time above 1300°C (hours)	Time at 1520±6°C (hours)	Porosity	Grain Size	Average Hardness of Ends* R_A(60Kg) Soft End	Hard End	Specific Gravity	Compression Strength (psi)	Remarks
CE27Co10-II 71	1 1/2	1	A-3	2-3,+7	89.0	89.3	14.31	523,000	
CE28Co10-II 71	1 1/2	1	A-3	2-3,+9	89.6	89.9	14.30	619,000	
CE29Co10-II 71	1 1/2	1	A-2	2-3,+6	89.1	89.5	14.34	552,000	For 1 hour sinter
CE30Co10-II 71	1 1/2	1	A-1	2-3,+7	88.5	89.5	14.35	608,000	Averages Comp. Str. = 594,000
CE31Co10-II 71	1 1/2	1	A-2	2-3,+6	89.5	89.5	14.31	***	Average Spec. Grav. = 14.32
CE32Co10-II 71	1 1/2	1	A-2	2-3,+8	89.2	89.2	14.32	670,000	
CE33Co10-II 73	2 1/3	2	A-3	3-4,+8	87.8	88.2	14.23	254,000	
CE34Co10-II 73	2 1/3	2	A-3	3-4,+7	88.3	88.6	14.34	566,000	
CE35Co10-II 73	2 1/3	2	A-1	2-3,+13	88.6	88.6	14.37	538,000	For 2 hour sinter
CE36Co10-II 73	2 1/3	2	A-1	3-4,+15	88.6	89.7	14.32	361,000	Average Comp. Str. = 548,000
CE37Co10-II 73	2 1/3	2	A-3	3-4,+24	88.3	88.9	14.31	541,000	Average Spec.Gr. = 14.31 (neglecting CE33Co10-II 73 and CE36Co10-II 73)
CE38Co10-II 73	2 1/3	2	A-2	3-4,+27	87.5	88.6	14.30	***	
CE39Co10-II 75	4 2/3	4	A-2	3-4,+18	86.5	87.1	14.33	460,000	
CE40Co10-II 75	4 2/3	4	A-2	3-4,+27	85.7	86.7	14.29	***	
CE41Co10-II 75	4 2/3	4	A-2	3-4,+30	86.6	87.1	14.27	488,000	
CE42Co10-II 75	4 2/3	4	A-2	3-4,+20	86.9	86.9	14.30	467,000	For 4 hour sinter
CE43Co10-II 75	4 2/3	4	A-2	3-4,+27	87.1	87.4	14.31	502,000	Average Comp. Str. = 471,000
CE44Co10-II 75	4 2/3	4	A-2	3-4,+22	86.7	87.0	14.31	439,000	Average Spec. Gr. = 14.30
HP20Co10-II 41	**	**	A-1	1-2	90.5	90.6	14.43	697,500	
HP24Co10-II 40	**	**	A-1	1-2	89.9	90.8	14.43	617,000	

* Ends ground until hardness was same after successive grindings

** Hot pressed 10 minutes at 1400 - 1425°C, 1500 psi

*** Porosity checked on longitudinal Section

TABLE IX

Showing Properties of Hot Pressed Compression Test Specimens
Made from Wolfram Carbide and Various Binder Elements

Specimen	Pressure During Hot Pressing (psi)	Temperature During Hot Pressing (°C)	Powder Composition	Milling Time (hours)	Specific Gravity	Hardness R_A(60Kg)	Compression* Strength (psi)
HP12Cr8 1/4 - 355	8000	1600	8 1/4%Cr, 91 3/4%WC	4 1/3	13.9	93.7-94.4	Broke during grinding
HP13Cr8 1/4 - 357	8000	1600	8 1/4%Cr, 91 3/4%WC	4 1/3	13.9	93.8-94.4	489,100
HP29Ni8 1/4 - 359	3500	1400	8 1/4%Ni, 91 3/4%WC	1 1/3	15.4	91.8-92.5	575,200
HP30Ni8 1/4 - 361	3500	1400	8 1/4%Ni, 91 3/4$WC	1 1/3	15.3	91.8-92.4	543,500
HP13Fe10 - 367	4000	1275	10%Fe, 90%WC	4 1/3	14.3	91.5-92.6	634,100
HP14Fe10 - 369	4000	1275	10%Fe, 90%WC	4 1/3	14.3	91.6-92.3	643,100
HP15Fe10 - 375	4000	1400	10%Fe, 90%WC	4 1/3	14.6	91.3-91.9	625,000
HP16Fe10 - 377	4000	1400	10%Fe, 90%WC	4 1/3	14.8	92.0-92.4	712,900
HP17Fe10 - 379	6100	1400	10%Fe, 90%WC	4 1/3	15.0	92.5-93.4	649,500
HP18Fe10 - 381	6100	1400	10%Fe, 90%WC	4 1/3	15.1	92.4-93.4	703,800

* Original data and plots of data are on file with Mr. K. Abbott at Watertown Arsenal Laboratory

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