SINTERED DIAMOND COMPACTS USING COBALT AS A BINDER

by

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This research was supported in part by the Air Force Office of Scientific Research under Grant AF-AFOSR-1255-67 Mod. C and by the National Aeronautics and Space Administration (Grant NGR 05-007-003).

ABSTRACT

Diamond powder can be successfully cemented with cobalt. At 62kb the sintering occurs over the range 1570° to 1610°C. The maximum microhardness (> 3000 kg/mm^2 on the Knoop scale) is obtained with 20% by volume of cobalt and a diamond particle size of 1-5μ.

Recently, Stromberg and Stephens[1] and H. T. Hall[2] have reported synthesizing a polycrystalline diamond compact, carbonado, at high pressures and temperatures. We have discovered a method for sintering diamond powder (also at high pressures and temperatures) using cobalt as a binder, analogous to its role in cemented tungsten carbide. In comparison with the published reports[1,2] on synthetic carbonado, it appears that this new material is easier to make and can be made in larger, more nearly uniform speciments. It is harder than cemented tungsten carbide and thus promises to be useful in many scientific and industrial applications as a replacement for it. We will describe the sintering conditions used and some of the properties of the cemented compacts obtained.

The starting materials were commercially available diamond powder of either 0-2μ, 1-5μ or 10-20μ size and cobalt powder of 0-5μ size and 99.9 + % purity. The powders were dried, weighed and mixed and then packed into tantalum containers for sintering. Pressure was generated in a piston-cylinder apparatus with appropriate modifications to allow us to work in the 60-65kb range[3,4,5]. The samples were heated by means of an internal graphite heater and the temperature was monitored with a Pt-Pt 10% Rh thermocouple. The thermocouple emf was corrected for the effect of pressure[6] and the correction corresponded to an addition of 15°C to the temperature values obtained directly from the standard tables.

We chose 62kb as a convenient pressure at which to work and then determined experimentally the temperature range in which the sintering occurred. Samples had to be heated above the cobalt-diamond eutectic temperature, but not so high as to graphitize the diamonds. At 62± 1kb the samples showed partial graphitization when heated above 1610° ± 10°C. This temperature is well below the 1700°C which is reported as the diamond-graphite equilibrium temperature at 62kb by Bundy, et al[7] and calculated by R. Berman[8]. Recently, John Haygarth[9] has used piston-cylinder apparatus similar to ours to determine a diamond-graphite equilibrium point at 51.8± 0.2kb and 1335°C. Using this point and Berman's slope[8] of 30.4°C/kb, one can calculate an equilibrium temperature of 1645°C at 62kb which is much closer to the value that we find. Both Stromberg and Stevens[1] and H. T. Hall[2] report working at higher temperatures at similar pressures to sinter diamonds, but they have no cobalt present to dissolve and reprecipitate the metastable diamonds as stable graphite and can therefore use higher temperatures. Below 1570°± 10°C, which is apparently the cobalt-diamond eutectic temperature at 62kb, no strong compacts were formed.

Therefore, 1590°C and 62kb were chosen as the sintering conditions. The samples were maintained at these conditions for about 20 minutes and were then cooled to room temperature in about an hour. As the samples were cooling, the pressure was slowly released in order to minimize the residual strains in the product but care was taken so that the samples were always kept within the diamond stability region.

The sintered samples were grayish, metallic looking, slightly ferromagnetic cylinders, ~6.3mm diameter and 2.5 to 9.0 mm long. Their properties were determined by X-ray diffraction, scanning electron microscope, electron microprobe, density and Knoop microhardness measurements.

The microhardness results are shown in Table 1 for various sintered samples. The values are averages for indentations over one or more polished cross-sections of a given sample. The relatively large deviations are a result of the difficulty in measuring the small indentations. The indentations were made with a force of 2kg and were about 100μ in length. Since the grain size of the samples is much smaller than 100μ these values are believed to reflect the hardness of the compact, not the hardness of only the diamonds. For comparison, the Knoop microhardness of diamond is 7000 kg/mm^2 [10], cubic BN has a value of 3800 kg/mm^2 [11] and cemented WC varies from 1400 to 1800 kg/mm^2 [10], depending on the amount of cobalt binder present. We tested two samples of cemented WC and the results are also shown in Table 1.

The samples containing 20% by volume of cobalt could be polished on fine-grained diamond wheels to a good metallic luster. Microscopic examination showed no signs of any diamond particles torn from the matrix during polishing. One could see the polished surfaces of the individual diamond grains indicating that they were indeed bound by the cobalt. Other indications of the strength of the material were that the polished surfaces could not be scratched with a tungsten carbide scribe and also that the Knoop diamond indentor required replacement after about 20 indentations into these surfaces.

The electron microprobe and scanning electron microscope showed that the diamond particles were well distributed in the cobalt matrix and that some diamond grain growth had occurred. A microprobe photograph of a sample containing 20% cobalt and starting with 1-5μ diamond powder is shown in Figure 1. In the photograph, the darker areas are cobalt and the lighter ones are diamond. There were some diamond grains with diameters as large as 6-8μ in the samples in which the starting diamond powder was 1-5μ. Also many diamond grains appeared to be fused together into clusters. These results suggest that the cobalt cleans the diamond surfaces of any adsorbed gases that might prohibit or retard grain growth or fusion, thereby making unnecessary the surface cleaning and degassing procedures described by Stromberg and Stephens[1]. The grain growth very likely occurs when some of the diamond particles dissolve in the cobalt and then reprecipitate on other grains, indicating that the molten cobalt wets the diamond surfaces.

As can be seen from Table 1 the optimum amount of cobalt for "cementing" the diamond powder is 20% (± 1%) by volume. Microscopic examination of samples with less cobalt content showed regions of unwet diamond particles even after sintering for 2 hours indicating that 20% cobalt is needed to wet all of the diamond surfaces. Samples with greater than 20% of cobalt are softer. An attempt to substitute nickel for cobalt under identical sintering conditions also led to a softer material.

The diamond particle sizes of 1-5μ and 0-2μ appear to result in harder compacts than the 10-20μ size particles. In one run we replaced ~ a quarter of the 1-5μ diamond powder with graphite powder and it was entirely converted to diamond under the sintering conditions leading to a large and non-uniform increase in the diamond grain size. It appears that more graphite can easily be substituted for diamond but that the resulting compacts may be a little softer due to the larger and less uniform diamond grain size.

The X-ray diffraction patterns on the samples with cobalt showed the presence of only fcc or β-cobalt which is stable above 450°C at atmospheric pressure[12]. No hcp or α-Co reflections were present. The density of each sample was found to be at least 98% of that of the theoretical density and most samples had densities greater than 99%.

In order to determine how well our new material would perform as a cutting tool and as a "truer" for grinding wheels, we did the following test. We weighed both our compact and a silicon-carbide, resin-bonded grinding wheel and used the compact to turn off some silicon carbide from the grinding wheel. We then reweighed both and determined a ratio of the weight losses. For a comparison, we also tested a commercial diamond truer and a piece of tungsten carbide under identical conditions. The weight-loss ratios for the diamond truer, our compacts with 20% Co and 1-5μ diamond powder, and tungsten carbide (10% Co) were 18,000:1, 9,500:1, and 1.5:1 respectively. Since the compacts with 20% by volume of cobalt contain only ~ 60% by weight of diamond, the compacts perform about 85% as well as pure diamond on a weight for weight of diamond basis.

In conclusion, we have synthesized a very promising new material which should have many commercial possibilities; for example, as a cutting tool or as a "truer" for grinding wheels. The procedure for its production is relatively simple and is economically feasible since inexpensive diamond dust along with graphite may be used as starting materials.

ACKNOWLEDGEMENTS

We thank Lee Ounanian and Charles Bayer for expert assistance with the experimental apparatus, J. C. Haygarth, I. C. Getting and G. C. Kennedy for much information and many helpful suggestions, R. Jones for use of the electron microprobe and W. Schopf for use of the electron microscope. The press used was financed by NASA Grant No. 287-62.

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TABLE 1

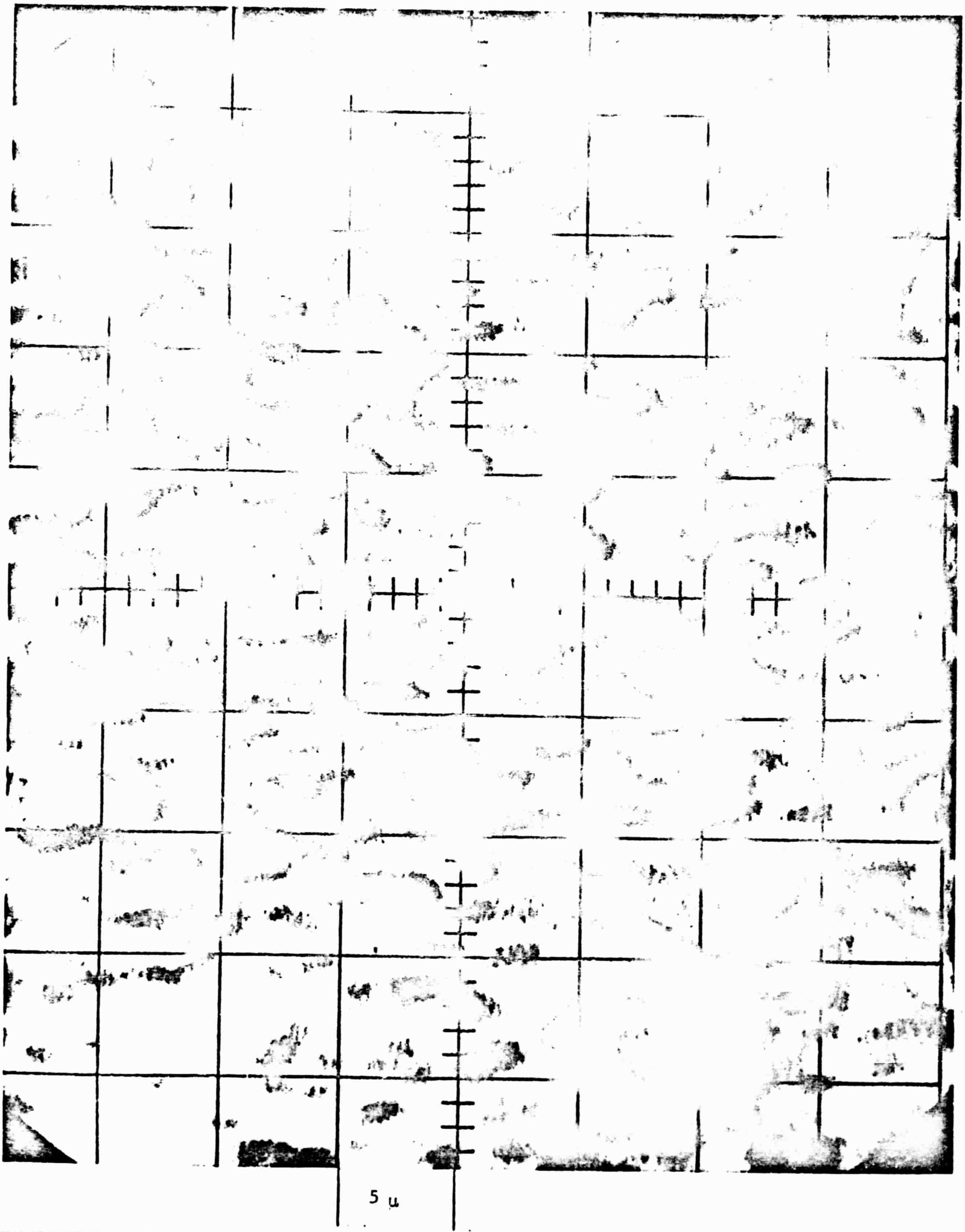
Knoop Microhardness of Various Samples

STARTING MIXTURE	KNOOP MICROHARDNESS (Kg/mm^2)
Co 20%*, Diamond 1-5μ	3000 ± 300
Co 20%, Diamond 1-5μ	2900 ± 300
Co 20%, Diamond 0-2μ	2900 ± 300
Co 20%, Diamond 1-5μ 60%, Graphite 20%	2800 ± 400
Co 20%, Diamond 10-20μ	2700 ± 100
Co 25%, Diamond 10-20μ	2200 ± 200
Co 16%, Diamond 1-5μ	2100 ± 200
Co 12%, Diamond 1-5μ	1600 ± 100
Ni 20%, Diamond 1-5μ	2200 ± 200
Tungsten Carbide - 15%* Co	1700 ± 50
Tungsten Carbide - 20% Co	1500 ± 50

*by volume (± 1%)

FIGURE LEGEND

Electron Microprobe photograph of sample containing 20% cobalt and 1-5μ diamond grains.


5 μ