A. A. GIARDINI AND J. E. TYDINGS

TABLE 1. OBSERVED	MINIMUM	PRESSURES	AND	TEMPERATURES	OF	DIAMOND
For	MATION FO	R SOME ME	TAL-	CARBON SYSTEM	s	

System	Pressure ¹ (kilobars)	Temperature ² (° C.)	Hold Time (Minutes)	
"Inconel"+Graphite	45	11.50°	2	
Mn, Cu (12/1 wt. ratio)+Graphite	48	1400°	2	
Cobalt+Graphite	50	1450°	2	
Mn, Co (12/1 wt. ratio)+Graphite	50	1450°	2	
Mn, Ni (12/1 wt. ratio)+Graphite	53	1475°	2	
Nickel+Graphite	55	1460°	2	
Pt, Co (4/1 wt. ratio)+Graphite	55	1500°	2	
Iron+Graphite	57	1475°	2	
Manganese+Graphite	57	1500°	2	
Tantalum+Graphite3	65	1800°	2	
Platinum+Graphite ³	70	2000°	2	
Chromium+Graphite ³	70	2100°	2	

¹ The stated pressures are those measured at room temperature prior to elevation of temperature. Measurements are based on the electrical resistance values of the pressure induced transitions of 0.5 mm diameter bismuth and barium wires encased in 3 mm diameter AgCl. Reference values of 25.4, 26.9, 60 and 90 kilobars have been used. Margin of error is believed to be approximately 1% at 30 kilobars, 3% at 50 and 5% at 70 kilobars.

² Temperatures at the location of diamond formation are believed to be correct to within 5% at 1500° C. and within 10% in the vicinity of 2000° C. Thermocouples of Pt-Pt (13% Rh) have been used.

³ The geometry of reactants used in these experiments was that of an elongated metal rod enveloped by a graphite sleeve. Some nickel contamination was experienced due to diffusion from the electrical contact disk (Fig. 3). The remaining experiments consisted of a vertical stacking of metal graphite-metal solid right circular cylinders.

regular microstructure is fully different from both the residual bulk unreacted graphite and that in contact with but undissolved in the metal. Figure 7(c) is an enlarged portion of 7(b). Figures 7(d), 7(e) and 7(f) show specimens of essentially three dimensional single crystal recrystallized graphite observed in pure platinum. The latter was reacted with graphite at approximately 2000° C. at 80 kilobars. No diamond was formed. X-ray diffraction of reaction products showed only graphite and elemental platinum. Diamond has been readily synthesized with platinum containing small amounts of nickel or cobalt. The latter tend to reduce temperature, and therefore, pressure requirements.

The reaction mechanism in carbide forming solvents constitutes a sequential formation of carbon compounds with increasing stoichiometric carbon content.¹ The sequence proceeds, in the presence of excess carbon, to the formation of an "ultimate carbide" having the highest possible

¹ Determined by analysis of residual products.

6



DIAMOND SYNTHESIS

INCREASING TEMPERATURE WITH EXCESS CARBON PRESENT

FIG. 8. A flow diagram illustrating the sequential nature of metal-carbon interaction in an excess of carbon) under high pressure as a function of temperature. The designated presence of Fe₂C is tentative.

stoichiometry of carbon. The latter, again in the presence of excess carbon, decomposes to the next lower carbide with the liberation of free carbon atoms. The chemistry is regenerative by reaction of the "lower carbide" decomposition product with additional unreacted graphite. The sequence has been observed both for pure iron¹ and manganese,¹ as well as chromium and tantalum both containing small amounts of nickel. Nickel was added to the latter two so as to reduce PT requirements to a more readily manageable range.

In view of the mechanism described above, the reaction philosophy of

¹ The "ultimate carbides" observed in the iron-carbon and manganese-carbon systems for the P-T values covered correspond to FeC and MnC, respectively.

1406

1407