

Vacuum Carburizing of P/M Steels

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Editor's Note: While there is information in the literature regarding conventional atmosphere carburizing and carbonitriding of P/M steels¹⁻⁵ other than work published by Weber⁶, there has been little information available regarding vacuum (partial pressure) carburizing of P/M steels. Presented in this article, which is from a paper delivered during 1987 Annual Powder Metallurgy Conference (sponsored by MPIF and APMI), is experimental data on vacuum carburizing P/M steels in the 6.8 to 7.3 g/cm³ (0.2 to 0.26 lb/cu in.) density range at 1550°F/843°C and 1700°F/927°C. The data was generated using a method based on previous work by F.E. Harris and C. I. Hayes, Inc. for wrought steels.

In atmosphere carburizing the gases surrounding a workload are a mixture of carbon monoxide, carbon dioxide, hydrogen, nitrogen, water vapor, and oxygen together with an enriching gas, usually methane or propane. Ammonia is added if carbonitriding is desired. The carbon potential of the atmosphere or driving force for carburization is dependent on the ability to balance these constituents under non-equilibrium conditions. If not properly controlled, carburizing/decarburizing (reducing/oxidizing) reactions can cause much slower rates of carbon absorption⁷. In addition, care must be taken to minimize dealloying at the surface of the steel which has been found to occur during atmosphere carburizing⁸.

Vacuum carburizing, can be thought of as a method of "pure" carburization and "pure" diffusion. The very nature of a vacuum environment makes it ideal for case hardening since it atomically conditions the surface of the steel prior to the start of the process. This eliminates trapped gases and volatile foreign material that would otherwise deter carbon absorption. Vacuum allows the desired chemical interactions to take place unhindered. It has been proven⁹ that vacuum carburizing al-

lows the steel surface to rapidly reach high carbon concentrations up to limits of solubility of carbon in austenite. Significant increases in mechanical properties can result from use of vacuum⁹.

Among the factors which influence and control the carburizing process are time, temperature, and carbon potential. In powder metallurgy, density considerations also must be included. F. E. Harris¹⁰ established an empirical atmosphere carburizing formula relating the principle wrought metallurgy variables (see Table I). Similarly, work done by C.I. Hayes, Inc.^{11,12,13} developed these relationships for vacuum carburizing (see Table II).

Extensive vacuum carburizing field results have been correlated with the established formulas for carburizing rates. Constants of proportionality have been shown to properly express how the case depth varies as the square root of time for various temperatures. These relationships can be conveniently expressed in graphical form (see Figs. 1 and 2 for 1550°F/843°C and 1700°F/927°C data). Thus, wrought material's case depths can be predicted with confi-

Table I "k" Values for Harris Formula

Temperature (°F)	k
1550	.0147
1600	.0177
1700	.0250
1900	.0458

"k" values based on Empirical Harris Gas Carburizing Formula:

$$T.C.D. = k\sqrt{t}$$

where: T.C.D. = Total case depth, in inches
k = Gas carburizing constant, temperature related
t = time, in hours

Table II "k₁" Values for Hayes Formula

Temperature (°F)	k ₁
1500	.0109
1600	.0128
1700	.0221
1900	.0442

"k₁" values based on Empirical Hayes Vacuum Carburizing Formula:

$$E.C.D. = k_1\sqrt{t}$$

where: E.C.D. = Effective case depth, in inches
k₁ = Vacuum carburizing constant, temperature related
t = time, in hours

dence and accuracy. Logically, an empirical relationship also can be developed to relate case depth and time as a function of density for

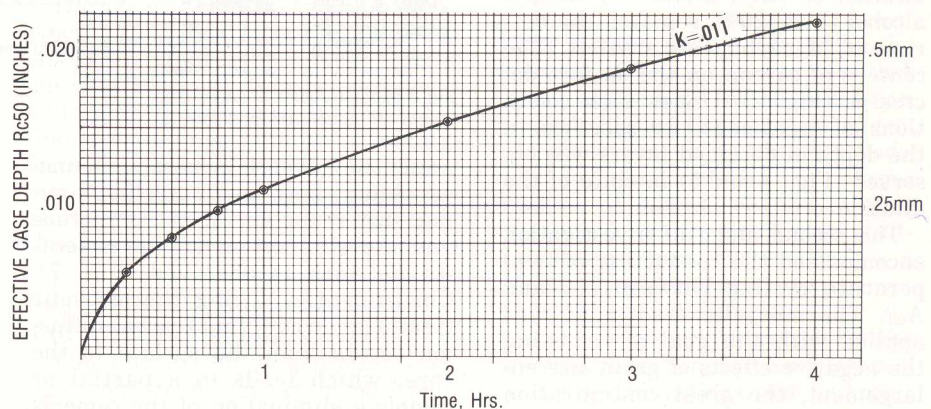


Fig. 1 Total time hours (carburize or carbonitride and diffusion)-1550°F.

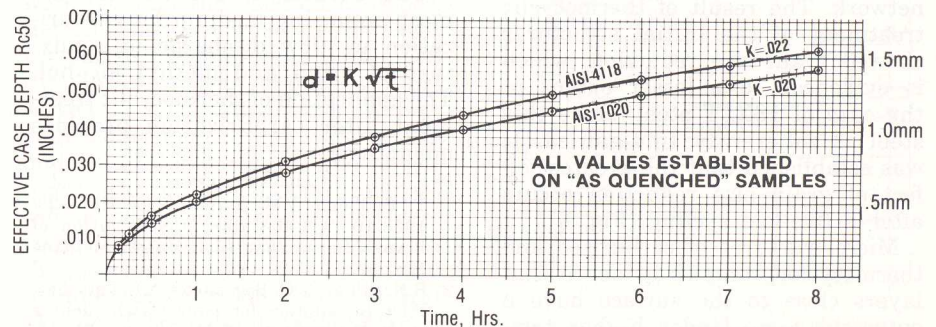


Fig. 2 Total time hours (carburize & diffusion)-1770°F (927°C).

P/M steels, as shown by the following work.

Experimental Procedure

Test pieces 2" (5cm) in dia. by 1" (2.5cm) thick were prepared from F-0002, FC-0202, and 4620 powders by Power-Tech Associates, Inc. to nominal "green" densities of 6.8, 7.0, and 7.3 g/cm³. The test pieces were delubed/presintered at 1550°F/843°C in an atmosphere of dissociated ammonia for 30 min. and then sintered in a vacuum furnace (Hayes CVPQ-091872) at 2250°F/1232°C for 60 minutes. The test pieces were gas pressure quenched with nitrogen at 40 psig to ambient temperature following sintering. A nitrogen partial pressure of approximately 200 microns was maintained during the sintering of the test pieces to minimize the vaporization of the copper in the FC-0202 material.

The test pieces were vacuum carburized in a furnace (Hayes VBQ-091824) with a propane partial pressure of 100 torr. All vacuum carburizing runs were preceded by a soak at the carburizing temperature of 60 min. and were followed by a quench in agitated, ambient temperature oil. The diffusion time was two thirds of the carburizing time for all cycles. The test pieces were subsequently degreased and tempered for 120 min. at 400°F/204°C in air. Table III details the four vacuum carburizing cycles used.

Following tempering, the test pieces were sectioned, mounted and prepared for metallographic examination and hardness traverses using standard techniques.

Hardness traverses were performed at 0.002 to 0.005 in./0.05 to 0.13 mm intervals normal to the diametral surface of the samples using a microhardness tester (Wilson) fitted

Table III Vacuum Carburizing Cycles Employed in P/M Tests

Cycle No.	Carb. Temp. (°F/°C)	Carb. Time (minutes)	Diffuse Time (minutes)
1	1550/843	30	20
2	1550/843	60	40
3	1700/927	30	20
4	1700/927	60	40

Table IV "k₂" Values for Hayes Formula (Includes Consideration of P/M Density)

Temperature (°F)	Density (g/cm ³)	k ₂
1550	6.8	0.026
	7.0	0.019
	7.3	0.006
1700	6.8	0.036
	7.0	0.024
	7.3	0.017

"k₂" values based on Empirical Hayes P/M Vacuum Carburizing Formula
 $E.C.D. = k_2 \sqrt{t}$

where: E.C.D. = Effective case depth, in inches
 k₂ = P/M Vacuum carburizing constant, temperature and density related
 t = time, in hours

with a Vickers indenter and a 100g load. The effective case depth (ECD) was determined by averaging the straight line portion of the hardness profile using a linear regression routine in a scientific calculator. From the regression coefficients, the distance from the surface at which a hardness of 513 VHN (R_c50) should be obtained was calculated.

Typically coefficients of determination (R²) of 0.90 or better resulted. Occasionally, with the lower density test pieces, the coefficients of determination were in the 0.80 to 0.90 range. This method of determining ECD has been found to be accurate and reproducible with both P/M and wrought steels.

The ECD's of the test pieces, along with the carburizing time at which they were produced, were "worked" through the Hayes equation ($D = k_1 \sqrt{t}$

where D is effective case depth in inches, k is a constant, and t is time in hours) to yield a "k" value. The k values for a given carburizing temperature and density were then averaged to give a "k₂" value.

With k₂ known, one can plot effective case depth versus total time (carburizing time plus diffusion time) for a given carburizing temperature and density and thus produce a convenient reference for estimating the carburizing process parameters necessary to develop a given ECD (see Figs. 3, 4, and Table IV).

Observations and Discussion

The surfaces of the test pieces were bright as-sintered. The F-0002 and 4620 materials at 6.8 and 7.0 g/cm³ (0.2 and 0.25 lb/cu in.) showed very little dimensional change from die size. At 7.3 g/cm³ (0.26 lb/cu in.) slight growth (0.2%) of the test pieces was observed. Significant growth (0.5 to 0.7%), which increased with density, was observed with the FC-0202 material.

The as-sintered microstructures exhibited well rounded porosity and uniform microstructures of fine lamellar pearlite and ferrite. A "white layer" (nitrogen rich) approximately 0.001 to 0.002 in./0.03 to 0.05 mm thick was observed adjacent to the surfaces of all the materials. This white layer formation is common with P/M materials pressure quenched with nitrogen directly from the sintering temperature.

The surfaces of the test pieces following vacuum carburizing were reasonably clean although a slight amount of carbon deposition was evident. The degree of carbon deposition decreased with increasing density and was more noticeable on the ends of the test pieces than on the sides. The large surface area of the

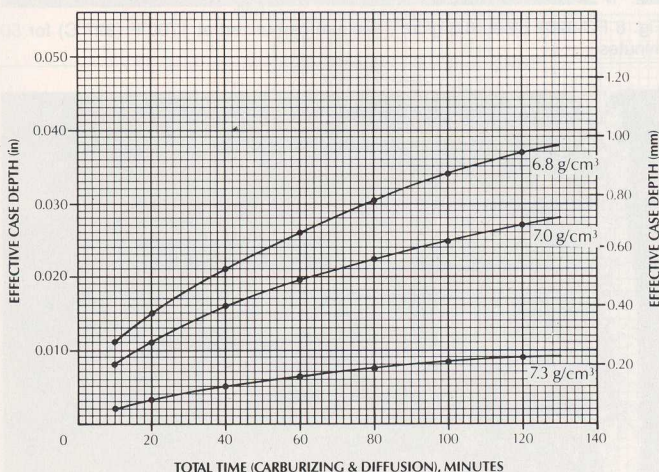


Fig. 3 Effective case depth vs. total time at 1550°F (843°C) for P/M densities shown.

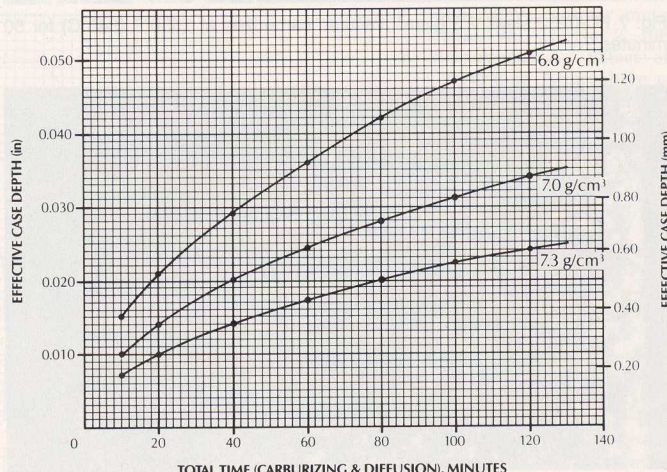


Fig. 4 Effective case depth vs. total time at 1700°F (927°C) for P/M densities shown.

P/M test pieces (as compared to the surface area of equivalent sized wrought test pieces) is believed to result in some thermal decomposition of the quench oil on the surfaces of the test pieces during the initial stage of cooling, and hence the slight carbon deposition observed.

The carburized microstructures produced at a given temperature varied principally as a function of density. At 1550°F/843°C, the materials at 6.8 g/cm³ exhibited predominantly plate type martensitic microstructures with approximately 10 to 20% retained austenite (see Fig. 5). The materials at 7.0 g/cm³ (0.25 lb/cu in.) exhibited mixed lath and plate type martensitic microstructures with little retained austenite evident (see Fig. 6).

The materials at 7.3 g/cm³ (0.26 lb/cu in.) exhibited predominantly lath type martensitic microstructures (see Fig. 7). At 1700°F/927°C, the materials at 6.8 g/cm³ (0.24 lb/cu in.) exhibited coarse plate type martensitic microstructures with 20 to 40% retained austenite (see Fig. 8). The materials at 7.0 g/cm³ (0.25 lb/cu in.) exhibited predominantly plate type martensitic microstructures with 10 to 20% retained austenite (see Fig. 9). The materials at 7.3 g/cm³ (0.26 lb/cu in.) exhibited mixed lath and plate martensitic microstructures with little retained austenite evident (see Fig. 10).

As one would expect, the hardness profiles also varied principally as a function of density. Typically, the maximum hardness increased, the ECD decreased, and the hardness profile became "steeper" with increasing density (see Fig. 11).

The variation in microstructure and thus hardness profiles with density is believed to be due to the increase in surface area and interconnected porosity combined with the decrease in the size of diffusion paths with decreasing density. At a lower density, there is a much greater surface area to volume ratio than at a higher density, thus carbon absorption to the limit of solubility occurs over a greater surface area yet

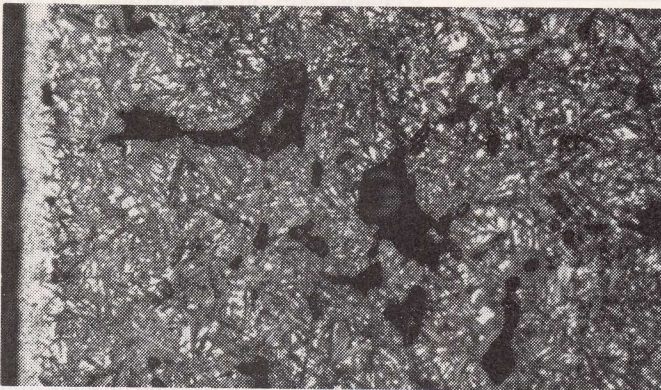


Fig. 5 FC-0202 steel, 6.8 g/cm³, vacuum carburized at 1550°F (843°C) for 50 minutes.

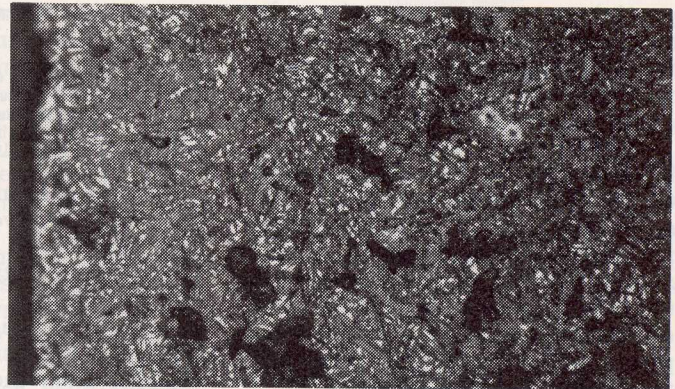


Fig. 6 FC-0202 steel, 7.0 g/cm³, vacuum carburized at 1550°F (843°C) for 50 minutes.

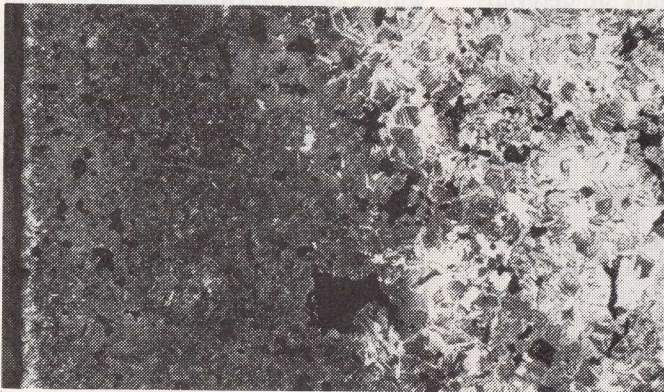


Fig. 7 FC-0202 steel, 7.3 g/cm³, vacuum carburized at 1550°F (843°C) for 50 minutes.

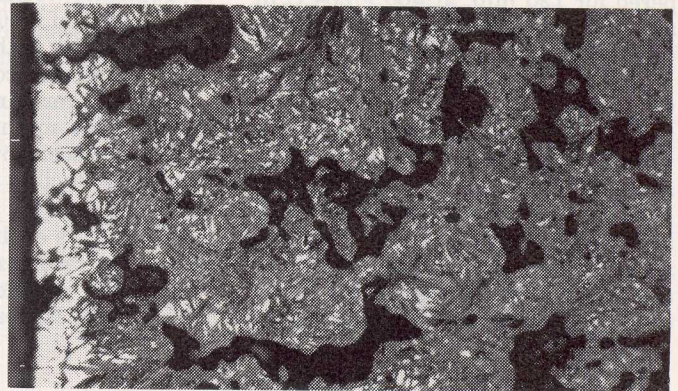


Fig. 8 FC-0202 steel, 6.8 g/cm³, vacuum carburized at 1700°F (927°C) for 50 minutes.

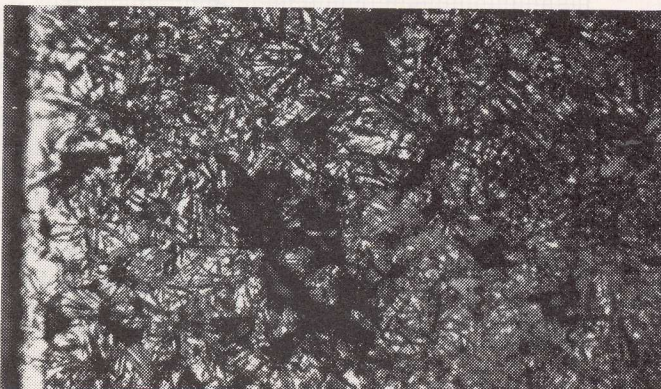


Fig. 9 FC-0202 steel, 7.0 g/cm³, vacuum carburized at 1700°F (927°C) for 50 minutes.

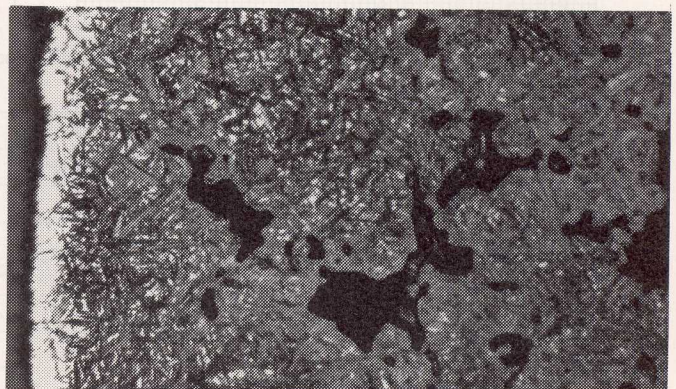


Fig. 10 FC-0202 steel, 7.3 g/cm³, vacuum carburized at 1700°F (927°C) for 50 minutes.

Table V K_2 Values for Conditions Shown

Carb. Temp. (°F/°C)	Density g/cm ³	Average k_2	Range of k_2
1550/843	6.8	0.036	0.031 to 0.043
1550/843	7.0	0.024	0.016 to 0.029
1550/843	7.3	0.017	0.014 to 0.019
1700/927	6.8	0.026	0.016 to 0.035
1700/927	7.0	0.019	0.012 to 0.025
1700/927	7.3	0.006	0.004 to 0.008

there is less volume of material to "hold" the carbon. This tends to result in a very carbon rich microstructure. Under the same conditions, a "leaner" microstructure would be expected with the same material at a higher density.

Table V details the average k_2 values at the two carburizing temperatures used.

As is apparent from Table V, the range about the average k_2 values is quite significant in some cases. This is believed to be due to the differences in the hardenability of the materials used in this investigation. The k_2 values presented above as average correlate with the FC-0202 material data quite well. The F-0002 material tended to yield lower k_2 values than above while the 4620 material tended to yield higher k_2 values.

The k_2 values presented would yield satisfactory results for an initial trial. The data from the first trial could then be used to calculate a k_2 value which would be more suited to the hardenability and surface characteristics of the material being used.

Summary

An empirical method for determining vacuum carburizing process parameters for P/M steels has been developed.

Plots of effective case depth versus total time (carburizing time and diffusion time) as a function of den-

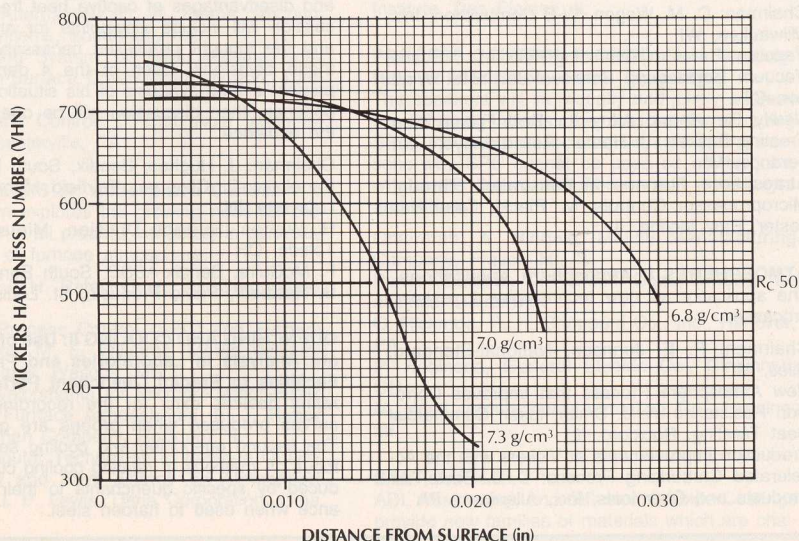


Fig. 11 Effect of density on case depth (1700°F).

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sity at 1550°F/843°C and 1700°F/927°C were developed. These plots can be used to establish initial vacuum carburizing process parameters for P/M steels in the 6.8 to 7.3 g/cm³ (0.24 to 0.26 lb/cu in.) density range.

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