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CARBURIZING OF STEEL USING "CRACKED" METHANE

BY

ROBERT A. HESSE

A THESIS

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PRESENTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE

OF

MASTER OF SCIENCE IN CHEMICAL ENGINEERING

ΑT

NEWARK COLLEGE OF ENGINEERING

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> Newark, New Jersey 1966

ABSTRACT

An attempt was made to carburize steel using cracked methane, industrial terminology for methane burned in air over a catalyst to give carbon monoxide and hydrogen, as the carburizing agent. Initial tests in a laboratory scale furnace using steel in chip form showed that the feasibility of this method was excellent. These tests also indicated that a good method of control of the carburizing would be by a system that could measure the dew point of the gases introduced into the fur-The next step was to apply the method of carburiznace. ing to small production size lots of bearing components. The first result was satisfactory. It was necessary to try to improve the surface carbon concentration to a more acceptable level since in this test only the lower limit was attained. Several more trials were run under varying conditions and limited success was met. Before the ideal results were obtained the furnace had a major breakdown and could not be repaired. Thus the project had to be discontinued.

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APPROVAL OF THESIS

CARBURIZING OF STEEL USING "CRACKED" METHANE

 $\mathbf{B}\mathbf{Y}$

ROBERT A. HESSE

FOR

DEPARTMENT OF CHEMICAL ENGINEERING

NEWARK COLLEGE OF ENGINEERING

BY

FACULTY COMMITTEE

APPROVED:

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NEWARK, NEW JERSEY JANUARY, 1967

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ACKNOWLEDGMENTS

The author wishes to acknowledge the help given him by the staff of the Metallurgical Laboratory of the New Departure - Hyatt Bearings Division of General Motors Corporation, and in particular, Mr. D. D. McCormack, whose encouragement convinced the author that the project could succeed, and also the advice of Dr. C. L. Mantell throughout the project.

iii.

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BACKGROUND

Since 1940, the practical inception date of gas carburizing of steel, the typical attitude of metallurgists has been that it is necessary to have a hydrocarbon gas as the carburizing agent. For purposes of easier reference and simplicity of terms, hereafter, a particular gas shall be discussed. The most commonly used gas for gas carburizing is methane found as the major constituent in refined natural gas. There are some traces of propane and butane present in the range of less than five percent each. It matters only slightly if methane is assumed as 100%. The principal concern is the pyrolytic destruction of a hydrocarbon such as:

> $CH_4 = C + 2H_2$ $C_2H_6 = 2C + 3H_2$ $C_3H_8 = 30 + 4H_2$

The final products are carbon and hydrogen in all cases. As long as a hydrocarbon or combination of hydrocarbons is used, the basic reaction of liberating carbon for diffusion into the steel is not affected. Therefore throughout this presentation a normal carburizing gas shall be called methane. Whether it is pure methane or some other combination is immaterial. This paper will deal with an effort to prove or disprove some of the original theories of gas carburizing as expostulated in 1940 and still accepted as fact. In a pamphlet prepared by the American Society for Metals entitled, "Furnace Atmospheres and Carbon Control", <u>ASM Monograph</u>. Metals Park, Ohio, 1964, the reactions that occur in a carburizing furnace are as follows:

Fe +
$$H_2O$$
 = FeO + H_2
Fe + CO_2 = FeO + CO
CO + H_2O = CO_2 + H_2
C + CO_2 = 2CO
CH₄ = C + 2H₂
Fe₃C + CO₂ = 2CO + Fe
3Fe + CH₄ = Fe₃C + 2H₂

The list is much longer if you include oxidizing equations of iron and even reduction equations of these oxides by hydrogen and/or carbon monoxide. Then equilibrium equations were written for most of these reactions, at least for those by manipulation of which the amount of carbon being made available to the system could be controlled. However, as early as 1941¹ it was stated that these equilibria constants are only good if the gas is passed very slowly through the furnace. Typical flow rates of gases through modern production furnaces are in the range of 300 to 400 cubic feet per hour. The furnaces have a volume of approximately 30 cubic feet when empty. Three-quarters of the volume is displaced by the work to be carburized, leaving less than 10 cubic feet of free space for the gases to react. This means that the gas in the furnace is completely changed at least 30 times per hour. This is not "very slowly" and the so-called equilibrium equations are not attained.

Metallurgists have, since the inception of gas carburizing, been trying to more effectively control the depth of carbon penetration, the carbon gradient through the carburized case, and condition of the surface of the carburized parts. F. E. Harris² in 1943 developed equations of carbon concentration and penetration at constant temperatures with respect to time. The case depth or depth of carbon penetration is determined by the following equation:

case depth =
$$31.6\sqrt{t}$$

10^(6700/T)

Where case depth is in inches, t is time at a temperature in hours, and T is the absolute temperature in degrees Rankine. For specific carburizing temperatures such as 1700°F. and 1750°F. the equation becomes:

case depth =
$$K\sqrt{t}$$

= 0.025 \sqrt{t} for 1700°F.
= 0.029 \sqrt{t} for 1750°F.

These two temperatures are mentioned as they are the usual carburizing temperatures at New Departure - Hyatt. To determine carburizing time, Harris developed the following equation:

carburizing time = total time
$$\begin{pmatrix} C - Ci \\ C_0 - Ci \end{pmatrix}$$

Where the total time is calculated in hours from the equation previously presented, C is the desired surface carbon concentration, C_0 is the surface carbon at the end of the carburizing cycle, and C_1 is initial carbon concentration of the piece before carburizing. The reason for this equation is that sometimes in order to obtain a desired case depth the surface carbon becomes excessive, and it is necessary to diffuse some of the carbon in the steel back into the atmosphere. This is done by maintaining the furnace temperature while stopping the flow of the carburizing gas.

In the article by Harris a mathematical explanation of the diffusion of carbon into steel was performed. The diffusion rate of the carbon was calculated by the Fick law of diffusion; which is the following differential equation:

$$\frac{\mathrm{dc}}{\mathrm{dt}} = \frac{\mathrm{d}}{\mathrm{dx}} \left(\mathbb{D} \frac{\mathrm{dc}}{\mathrm{dx}} \right)$$

where: c denotes carbon concentration at any given point

t denotes elapsed time

x is the linear distance of the given point below the surface

D is the diffusion constant

By manipulation of this equation the equation for case depth was derived and subsequently nearly all carburizing time tables used in the industry.

Practically, however, the time of carburizing is

not obtained so easily. A test sample is taken after the "proper" cycle has been completed and checked for accuracy. After checking carbon concentration through the test sample, the cycle is then adjusted to obtain the desired value. As in all other cases Harris assumed the diffusion rate is constant at any given point in the area being carburized. Since the driving force changes incrementally through the case, so must the diffusion rate. Harris admits there is a small error from this fact but then does not include it in his table of case depths for various times and temperatures. Figure 1 is a typical table of values as determined by the Harris Method.

Most metallurgists with the responsibility for carburizing steel believe that the operation must be carried out in the presence of a carburizing gas, meaning an uncracked hydrocarbon. However, it is possible to produce a hydrocarbon in the furnace without the presence of an uncracked hydrocarbon in the gas supplied to the furnace.

An important factor not discussed in any prior publication was brought to light in a procedure⁵ written for furnace operators for the addition of methane during carburizing. It pointed out that for more than one-half of the total cycle no methane should be added. However, some carburizing must be done during this time in which only

the cracked methane is added to the furnace. Procedural data is considered as a trade secret in reference to specifically constructed furnaces. Therefore it could not be included in this paper.

In the light of all these facts, it seemed possible that carburizing of steel could be effected without the addition of methane to the "cracked" carrier gas.

THEORY

It is proposed to carburize steel using only the "cracked" methane gas that is generated to be used as a carrier gas in most carburizing practices. This can be accomplished by utilization of the following reaction:

 $CO + 3H_2 \stackrel{N_2}{=} CH_4 + H_2O$ The methane produced then reacts most probably in the following manner:

$$\mathrm{CH}_{44} = \mathrm{C} + \mathrm{2H}_{2}$$

which is the accepted equation for producing available carbon for carburizing. It is the theory of the author that under the proper conditions this carrier gas can be used as the carburizing gas also. The carrier gas is a mixture of nitrogen, hydrogen and carbon monoxide. There may at times be traces of methane and carbon dioxide present, but this only occurs with improper control of the generators. The above reaction to produce methane has been known for nearly one hundred years. It was not until 1905, however, that the reaction was patented by Sabotier. Since then many industries have used this reaction to produce methane for consumption. However, it is still only used in the carburizing industry as a carrier gas and occasionally as an enriching agent for very small case depth increments.³

The peak conversion rate of carbon monoxide to

methane with a nickel or iron catalyst in the presence of nitrogen occurs between 1250°F. and 1450°F. A curve based on data from <u>Thorpe's Dictionary of Applied Chemistry</u>, Fifth Edition. New York: Longman's Green and Co., Inc., 1950, for the percent completion of this reaction is shown as figure 2 on page 23. Actually there is a variance of opinions as to where the peak of this curve can be located, and therefore the subsequent downward slopes vary also. In order to have some credence for this project, the data that corroborated the author's beliefs best were chosen as those presented. There is in the current data presented in various chemical encyclopedia a difference of more than 350°F. for the maximum value of conversion.

At 1750°F. the conversion should be 14%. This would mean that if 40 cubic feet per hour of the cracked gas were flowing through the furnace of which 25% was carbon monoxide, the amount of methane formed would be approximately one and one-half cubic feet per hour. This amounts to 0.075 pounds of methane per hour; further reduced to carbon available for diffusion, this amounts to approximately 0.055 pounds.

Normally in the carburizing of steel for the bearing industry, it is desired to attain a surface carbon value of approximately 1.00% starting with a base carbon of 0.20%. The penetration depths are dependent on the

carburizing time only. Figure 3 on page 24 shows a series of these desired curves. Assuming a straight line curve and an average value of 0.60% carbon for the entire depth, the average addition of 0.40% carbon is effected. This percent carbon is a weight percent. Therefore with an average addition of 0.004 pounds of carbon per pound of steel, a one hundred pound load in the furnace would require 0.4 pounds of carbon to carburize from a base carbon of approximately 0.20% to a surface carbon of nearly 1.00% through a 0.050 inches in depth. The one hundred pound load is used in these calculations because it is the standard load for the furnace used in this project.

If the breakdown of the methane produced were 100%, eight hours at 1750°F. would be the desired time cycle. At this temperature the methane breakdown should, however, be only a little over 90%. But still eight hours should be sufficient to carburize the steel to the desired depth and very close to the desired surface carbon.

During the regular production type carburizing, which was 40 cubic feet per hour of methane, based on the above calculations of methane necessary, the flow rate is so much in excess that it is unforseeable how this can be called controlled carburizing. No matter how poorly the furnaces are maintained, the material must reach the desired carbon impregnation levels when nearly 25 times the

necessary amount of methane is added to the furnace.

One other important factor to be considered in steel carburizing practices is the addition of carrier, or cracked methane, gas to the methane. The prevailing metallurgical theory is that this cracked gas adds to the carburizing potential of the total gas by the following equation:

$$200 = 0 + 00_{2}$$

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This reaction, however, is favored to the left above $1450^{\circ}F.^{6}$ A plot of this reaction is shown as figure 4, page 25. As previously stated, the reaction that can occur at the temperatures in question is that in which carbon monoxide is converted to methane in the presence of nitrogen and nickel or iron. This is the reaction that is utilized in this project.

Some of the other reactions that are currently presented as occurring during carburizing are also based on room temperature chemistry and do not really apply to carburizing. Therefore, rather than pursuing these equations and proving that they are invalid, the author attempted to prove that the carburizing of steel can be controlled accurately and scientifically rather than by trial and error.

EXPERIMENTAL PROCEDURE

In order to establish the feasibility of carburizing using the cracked methane, it was necessary to carry out several trial laboratory scale tests. A small furnace with a one inch diameter silicon tube as the furnace which was heated by electrical coils was used as the experimental model. The effective heated length of the tube was slightly more than six inches. This was determined by placing a platinum-rhodium thermocouple in the furnace and measuring the temperature at intervals of one inch along the tube after an apparent maximum temperature had been attained. Next a thermocouple was used to measure the uniformity of the temperature of long periods of time by keeping the thermocouple in one spot and taking readings at 15 minute intervals. Plots of these two temperature measurements appear as figures 5 and 6 on pages 26 and 27.

The discharge end of the furnace tube was plugged with a rubber stopper with a short glass tube, 1/4 inch diameter, through the stopper. The inlet side of the tube was outfitted in the same manner with the exception that the desired gas mixtures could be brought to the furnace by means of a rubber tube. A' drawing of the entire set-up is shown as figure 7 on page 28. The plugging of the ends of the tube was to preclude air from entering the furnace and upsetting the chemical balances that were trying to be attained during the experiment.

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The samples in the early laboratory determinations were in chip form as supplied by the National Bureau of Standards for a low carbon steel. The sample used was N.B.S. 111b with a carbon content of 0.21%. The chips were carefully weighed out to two grams and placed in a combustion boat cover into which a shallow layer of alundum had been spread. A drawing of the boat cover, alundum, and chips is shown as figure 8 on page 29. The sample was introduced through the discharge end of the furnace tube after the gas mixture had been flowing through the tube for a minimum of fifteen minutes. After the sample was in place, the stopper with the glass tube was reinserted and the exhaust gases through the tube were ignited. The ignition of the gases served a three-fold purpose: one - to assure the experimenter that the gases were flowing; two - to oxidize any noxious gases that might be in the system; and three - by noting the length of the exhaust flame, the desired gas flow could be maintained if the flame were of relatively constant length.

The first series of tests consisted of passing the cracked methane through the furnace and over the chips at 1750°F. for varying times to determine if a maximum carbon level could be reached. It was found that approximately 1.10% carbon was the maximum carbon content regardless of

time, figure 9, page 30. From this it was decided to determine what the controlling factors were that caused the maximization. The predominant theory in carburizing is that either the dew point control or the carbon dioxide must be controlled to effectively achieve carbon control.^{2, 4} Analyses of the gas effluent during the trials showed no traces of carbon dioxide. Therefore, the dew point of the exit gases was chosen as the first variable to be considered.

A water trap was placed in the entrance line of the furnace in which either a drying or wetting agent could be placed so that the dew point could be varied on either side of the as received condition. The dew point was varied from -40° F. to $+75^{\circ}$ F. Through the control of the dew point measured by both an Alnor dew point determining apparatus and a carbon dioxide cooled dew cup, the carbon content of the chips was varied from a high of 1.60% with the -40° F. dew point and a low of 0.08%, a loss of carbon, with the $+75^{\circ}$ F. dew point. These tests were all run for one hour duration. A test of eight hours at -40° F. dew point was run to see if any additional carburizing would take place. The results of these tests of dew point control are shown as figure 10, page 31.

Since carbon dioxide was also mentioned as a means of control of carburizing, the next series of tests dealt

with the inclusion of various amounts of carbon dioxide into the gas stream. The amount of carbon dioxide was varied from 0%, the as received gas, to 3.4% by obtaining special mixtures of gas from The Matheson Co., Inc. in East Rutherford, New Jersey. All gas samples were rechecked both before using them and as the effluent from the furnace tube for carbon dioxide on a gas chromatograph. As anticipated from theory, the increasing amounts of carbon dioxide lowered the amount of carbon that could diffuse into the chips, and at 3.4% CO₂ the carbon content of the chips was less than the original after carburizing. See figure 11, page 32.

Since it was possible to control the level of carbon in the chips, the next trial consisted of carburizing a small rectangular bar to determine if a carbon gradient through the bar were possible. The amount of surface area available was quite small compared to that of the chips; therefore, some difficulty was expected. No carbon pickup was discernible by the normal carbon analysis method. However, sectioning the bar lengthwise and etching in a 5% Nital solution, carbon penetration could be seen at the corners of the bar that were towards the incoming gas. Figure 12, page 33, is a schematic of this effect. From this it became evident that any further work to be done must be attempted in larger production type furnaces.

An AGF No. O furnace was chosen as it was the smallest production furnace available with facilities for supplying the "cracked" methane. The furnace had an interior volume of 0.75 cubic feet which could hold 100 pounds of work to be carburized if the pieces were not larger than three inches in diameter. Therefore, the amount of scrap produced in the trials would be kept to a minimum. Samples were taken of regular production and carbon gradient analyses were performed so that the experimental goal was to achieve the same carbon gradient as normal production. The inlet gas piping had to be rearranged with a by-pass to a drying column so that the dew point of the gas could be decreased. The dew point, however, could not be measured effectively in this furnace as both methods used in the laboratory scale tests were not accurate. The inaccuracy is because the temperature of the gas being sampled is too high to effectively measure. The results would be inconsistent according to the manufacturers' representatives in verbal communications with them. This proved to be true so that no dew point measurements were made during these production lot size trials. Carbon dioxide analysis was made from gas samples drawn out of the mid-point of the furnace chamber.

The initial test was run with a gas flow of 40 cubic feet per hour which is the maximum recommended flow rate

for this furnace. It was believed that this flow might be too high to allow for the reaction to take place. The duration of the test was eight hours, which was also the length of the carburizing cycle in the regular production run and the temperature was apparently maintained at 1750°F. according to the temperature recording instrument for this furnace. For the initial run the results were quite satisfactory with a surface carbon of 0.85% and an apparent case depth of .055 inches with an excellent carbon gradient, according to Mr. D. D. McCormack, Supervisor of Research and Development at New Departure - Hyatt Bearings Division, General Motors Corporation. The results of this test are shown as figure 13, page 34.

The only refinement necessary was to increase the surface carbon to approximately 0.95% as required in the regular production specifications. Since the dew point could not be measured, but was believed to be in the neighborhood of -40° F. as the indicating drying agent showed still more capacity for moisture absorption and the carbon dioxide content was undetectable, the gas flow was decreased to 20 cubic feet per hour with the belief that the rapid flow of gas through the furnace did not allow for complete reaction of the gases. The results of this test showed a decrease in surface carbon to 0.65% and case depth to .040 inches. Calculation of the amount of methane that

could be generated in the furnace proved that these results should have been expected. According to the percent of completion of the reaction, 14% at 1750°F. and 25% at 1650°F., the same reduced flow rate should produce enough methane to give the desired results. The test was run with the reduced temperature and the results were the same as the previous test with the exception that approximately 0.08% CO2 was found in the furnace. It was observed by the operator of the furnace that near the end of the cycle a leak had developed in the rear head of the furnace which could have accounted for the CO_2 found. There was no evidence of CO_2 in the cracked gas at the generating station. The head was repaired and the test repeated at a longer cycle only to repeat the results of the two previous tests. The next test consisted of increasing the flow rate to 30 cubic feet per hour and the temperature in the furnace to 1750°F. An increase of approximately 0.10% surface carbon was experienced. The final test was run at 40 cubic feet per hour and 1750°F. with a longer cycle. The results approximated the initial trial by yielding a surface carbon of 0.80 - 0.85%. No further tests were possible as the furnace completely broke down and it is not expected to be repaired until it is moved to its new location in Bristol, Connecticut, within the next two years.

It was hoped to run a test with increasing amounts

of the cracked gas until such a time as either the goal of 0.95% surface carbon was reached or a maximization of surface carbon occurred.

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The experimental data logs are shown as figure 14.

CONCLUSIONS

Since it was proved possible to carburize steel chips to a value of 1.60% carbon using the cracked methane as the only gas for the furnace atmosphere, it must also be possible to do the same with larger pieces of steel such as those used in the manufacture of bearings. The only problem that exists to date is the availability of furnace time. The method of arrival at the proper conditions for attaining satisfactory carburizing must be trial and error as the equilibrium equations involved are not pertinent to this type of reaction.

The design of the experiment was to utilize the cracked methane gas and to also prove or disprove some of the prevalent theories of gas carburizing of steel. It was shown that the most important factors of control are, as stated in prior work, dew point control and carbon dioxide control. Therefore it is believed that through the work in this experiment the following equations are the only ones necessary to maintain control:

1. $CO + 3H_2 = CH_4 + H_2O$ (above 1400°F.) 2. $2CO + O_2 = 2CO_2$

3.
$$CH_{\mu} = C + 2H_2$$

The other equations mentioned in earlier studies are all equations of what happens to the carbon liberated by the pyrolitic destruction of methane.

If the dew point is controlled, the amount of water in the system formed by equation No. 1 will be controlled thus shifting the direction of the reaction to whichever side is necessary. Similarly, if the amount of O₂ present is rigidly controlled, equation No. 2 will control the amount of carbon monoxide available for formation of methane. In the limited scope of this experiment, it was impractical to control the oxygen. Therefore dew point control was necessary and most useful. Carburizing furnaces today are controlled by either dew point levels or carbon dioxide levels. The dew point methods are the simplest and perhaps most effective. The carbon dioxide control is presently accomplished by means of infra-red analysis in most applications.

One other factor not included, heretofore, is that the material carburized with the cracked methane is much cleaner. That is to say, it is more free of scale or other carbonaceous material on the surface of the steel being carburized. This factor has been brought to the attention of several metallurgists at New Departure -Hyatt and they are in accord. However, they also noticed a factor which could be detrimental. By carburizing with this gas a phenomenon known as grain boundary oxidation has appeared when making metallographic observations of

cross-sections of the parts that were carburized. A possible chemical reaction for this phenomenon has been proposed as follows:

Fe + CO = FeO + C

However since the oxidation does not occur over the entire surface but only at areas that contain sharp corners, such as a relief groove at the base of a flange on bearing component, this reaction has not been proven as yet. This is really not a deterrent since many of the parts that are currently being carburized with the mixture of methane and cracked methane as a carrier gas exhibit the same structure, and these parts are acceptable by all current testing methods.

COMPARISONS OF TOTAL CASE DEPTHS

FOR DIFFERENT CYCLES AT HEAT

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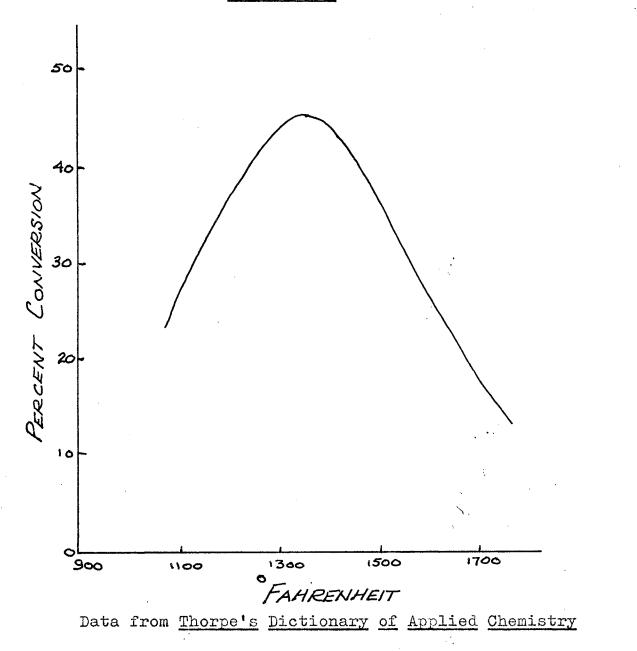
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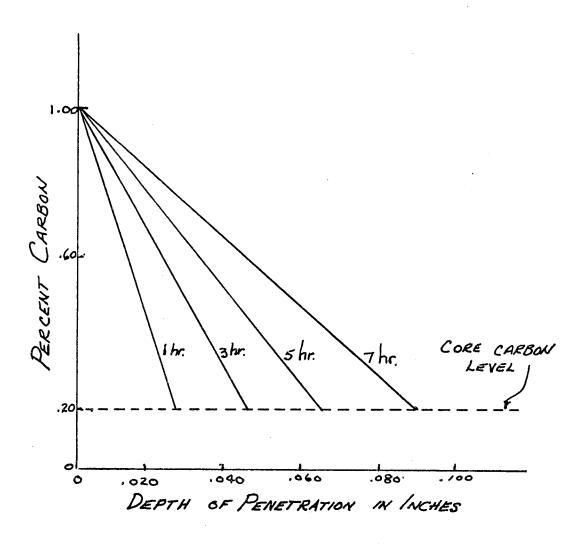
PERCENT CONVERSION CO TO CH4

vs.

TEMPERATURE



IDEALIZED CARBURIZATION CURVES



<u>REPRESENTAION OF EQUILIBRIUM BETWEEN CO & CO</u>2

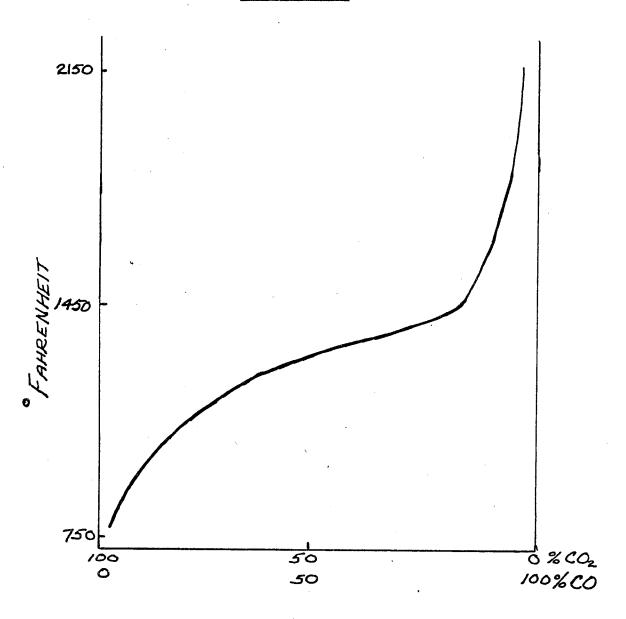
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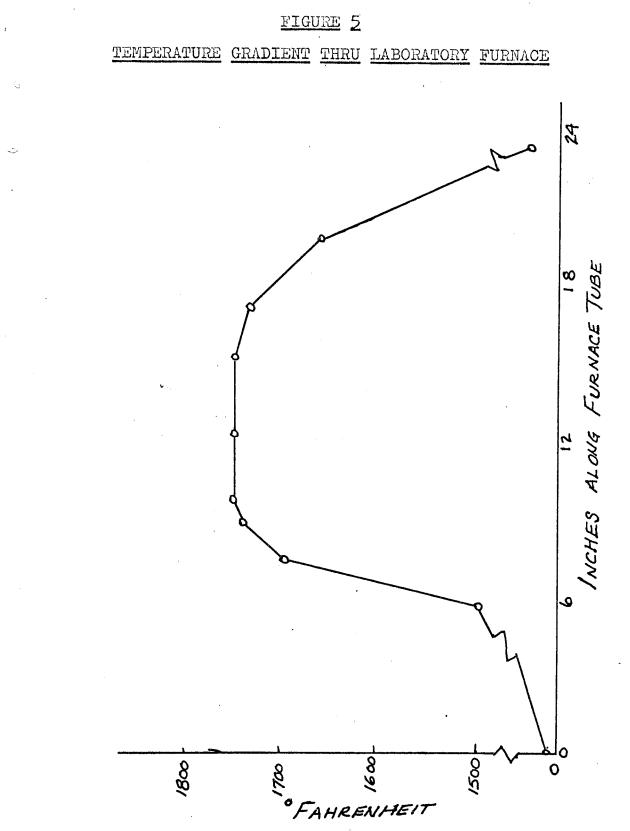
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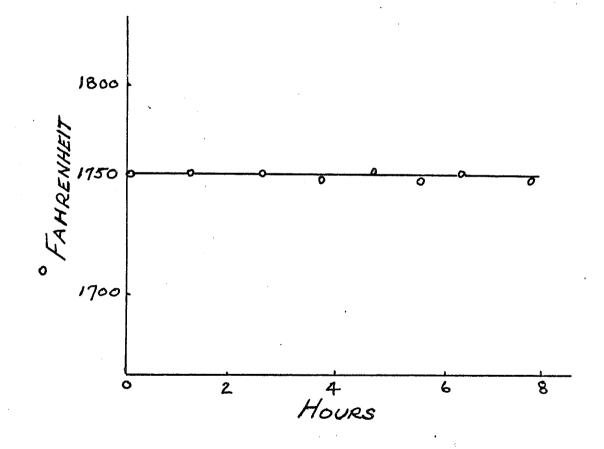


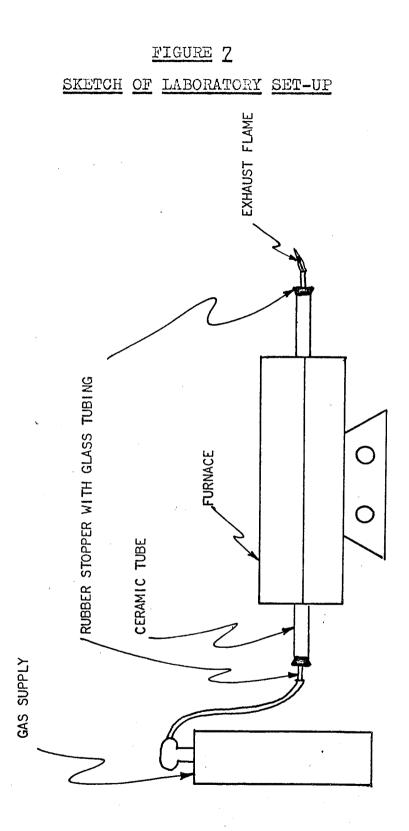


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TEMPERATURE UNIFORMITY OF LABORATORY FURNACE





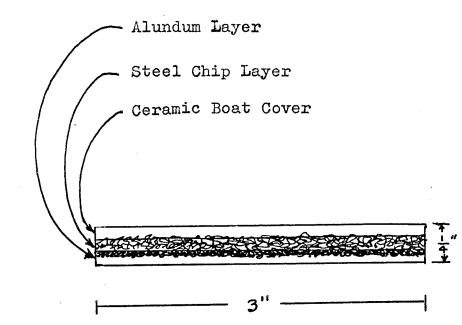
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SKETCH OF SAMPLE HOLDER FOR LABORATORY FURNACE



RESULTS OF FIRST SERIES OF LABORATORY TESTS

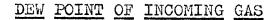
TEST NO.	TIME	% CARBON
1 ·	l hour	1.08
2	2 hours	1.10
3	4 hours	1.09
4	8 hours	1.11

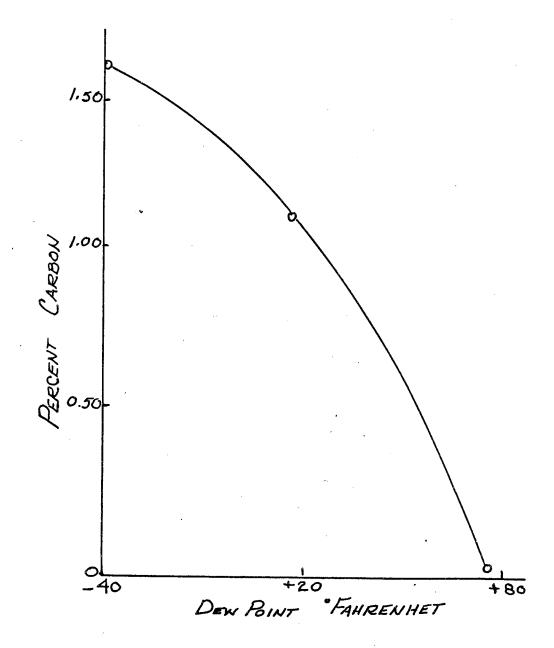
ALL TESTS AT 1750°F.

AMOUNT OF CARBON IN N.B.S. 1116 STEEL CHIPS

AFTER CARBURIZING

vs.

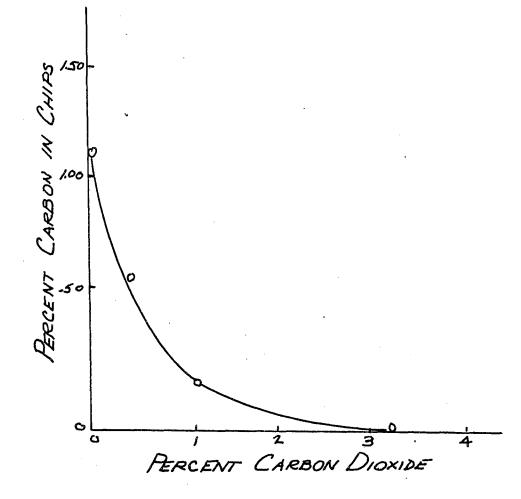




AMOUNT OF CARBON IN N.B.S. 111b STEEL CHIPS AFTER CARBURIZING

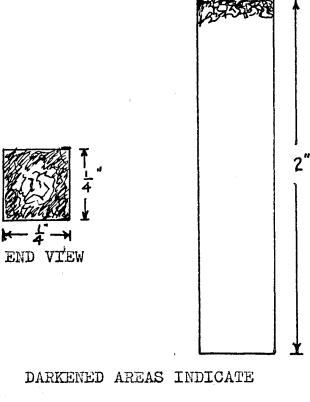
vs.

PERCENT CARBON DIOXIDE IN INCOMING GAS



3.

FIGURE 12 SCHEMATIC OF CARBON PENETRATION OF BAR IN LABORATORY FURNACE



SIDE VIEW

CARBURIZING HAS OCCURRED

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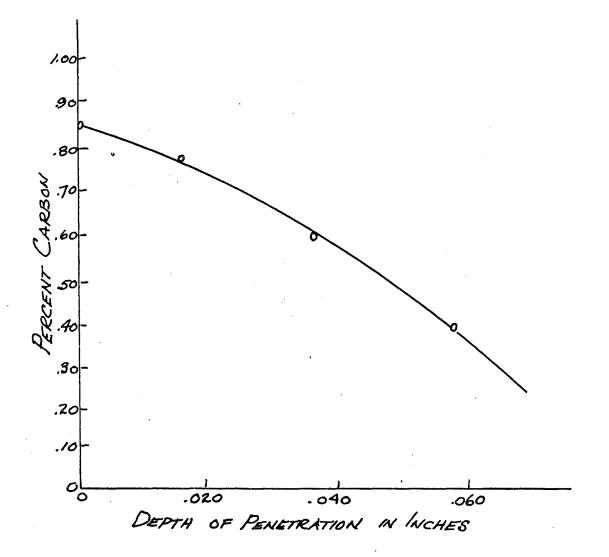
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CARBON PENETRATION IN PRODUCTION LOT TRIAL

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EXPERIMENTAL DATA

TEST	FURNACE	GAS_USED	TIME	CARBON_ANALYSIS
l	Lab.	CO, N ₂ , H ₂	l hr.	1.08
2	Lab.	CO, N ₂ , H ₂	2 hr.	1.10
3	Lab.	co, N ₂ , H ₂	4 hr.	1.09
4	Lab.	co, N ₂ , H ₂	8 hr.	1.11
5	Lab.	CO, N ₂ , H ₂ with Dew Point ² -40°C.	l hr.	1.60
6	Lab.	CO, N ₂ , H ₂ with Dew Point ² -40°C.	8 hr.	1.60
7	Lab.	CO, N ₂ , H ₂ with Dew Point ² +25°F.	l hr.	1.08
8	Lab.	CO, N ₂ , H ₂ with Dew Point ² +75°F.	l hr.	0.08
9 1	Lab.	$^{\rm CO}$, $^{\rm N}$, $^{\rm H}_2$ + 0.4 $^{\rm CO}_2$	l hr.	0.52
10	Lab.	CO, N ₂ , H ₂ + 1.0 CO ₂	l hr.	0.22
11	Lab.	$C0, N_2, H_2 + 3.4 C6_2$	l hr.	0.04
12*	Lab.	co, N ₂ , H ₂	l hr.	0.20
13	AGF No. O	CO, N ₂ , H ₂ 40 c.f.h. ²	8 hr.	Surf. carb. 0.85 Case depth .055
14**	AGF No. O	$C0, N_2, H_2$ 20 c.f.h. ²	8 hr.	Surf. carb. 0.65 Case depth .040
15	AGF No. O	CO, N ₂ , H ₂ 30 c.f.h. ²	8 hr.	Surf. carb. 0.73 Case depth .045
16	AGF No. O	CO, N ₂ , H ₂ 40 c.f.h. ²	8 hr.	Surf. carb. 0.82 Case depth .055
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ALL TEMPERATURES 1750°F.

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*This was the rectangular bar described in experimental procedure and effects shown as figure 12.

**Leak in furnace; 0.08% CO2 present.

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