US 20080149227A1

(19) **United States**

(12) **Patent Application Publication**

Connery et al.

(10) **Pub. No.: US 2008/0149227 A1**

(43) **Pub. Date: Jun. 26, 2008**

(54) **METHOD FOR OXYGEN FREE CARBURIZATION IN ATMOSPHERIC PRESSURE FURNACES**

(76) Inventors: **Karen Anne Connery**, Westmont, IL (US); **Robert Marcel De Wilde**, St. Niklaas (BE); **Richard A. Novak**, Naperville, IL (US); **Simon Ho**, Naperville, IL (US); **Amitabh Gupta**, Naperville, IL (US)

Correspondence Address:
PRAXAIR, INC.
LAW DEPARTMENT - M1 557
39 OLD RIDGEBURY ROAD
DANBURY, CT 06810-5113

(21) Appl. No.: **11/645,447**

(22) Filed: **Dec. 26, 2006**

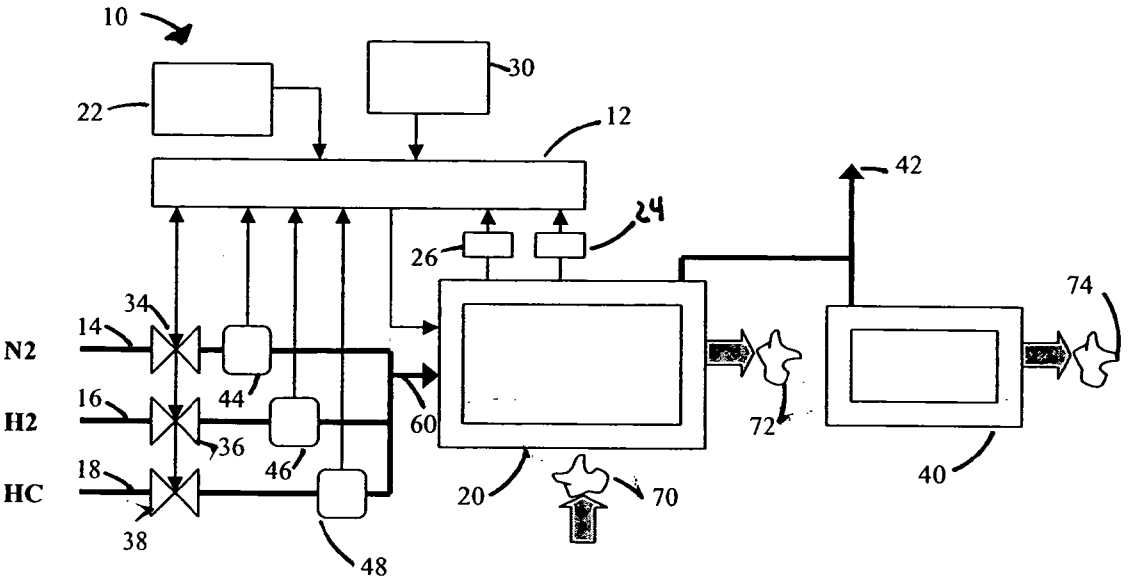
Publication Classification

(51) **Int. Cl.**
C23C 8/00 (2006.01)

(52) **U.S. Cl.** **148/223**

(57) **ABSTRACT**

A method of oxygen-free heat treatment of a steel part in an atmospheric pressure furnace is disclosed. The present method employs an oxygen-free controlled gas atmosphere including hydrogen gas in concentrations between about 1.0 percent to 10.0 percent, a hydrocarbon gas, such as propylene, in concentrations of between about 0.1 percent and 10.0 percent that varies as a function of time, with the balance of the gas atmosphere being nitrogen. The presently disclosed oxygen-free heat treatment process, preferably a carburization process, uses a precisely controlled atmosphere to minimize inter-granular oxidation, eliminate the formation of soot and cementite or other metallic carbides, and avoid hydrogen embrittlement.



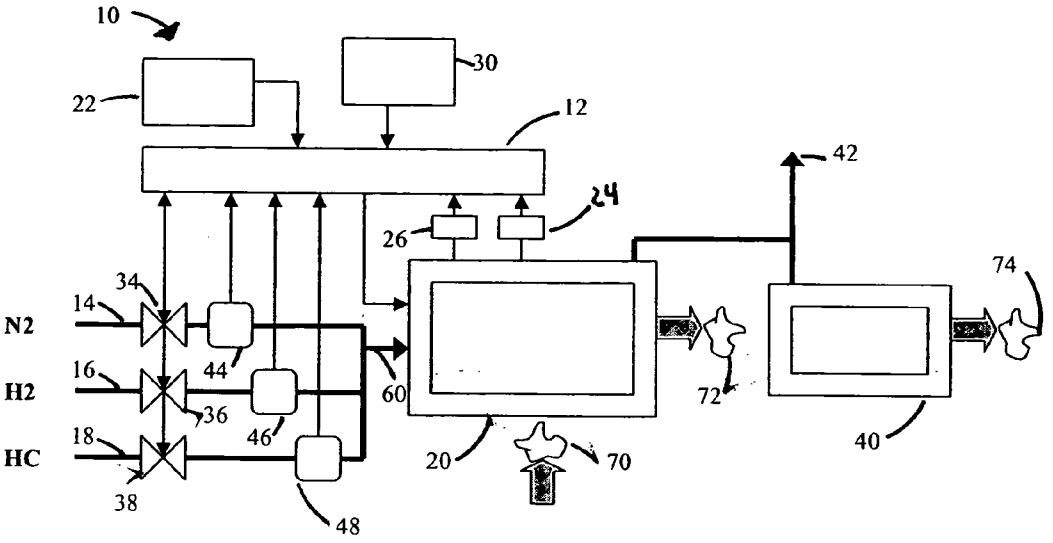

10
22
30
12
42
24
26
74
34
14
N2
16
H2
18
HC
44
36
46
38
48
60
20
70
72
40

Figure 1

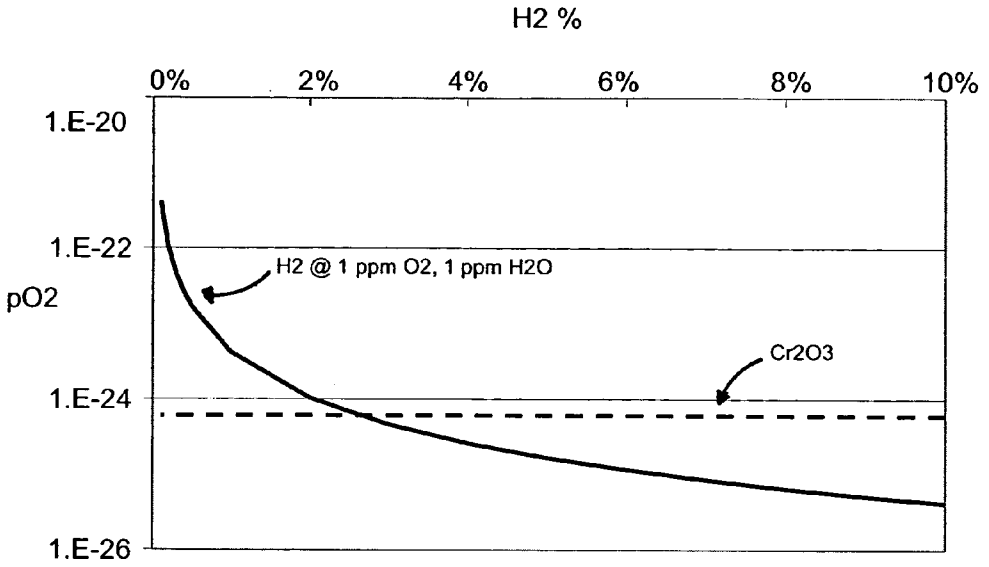


Figure 2

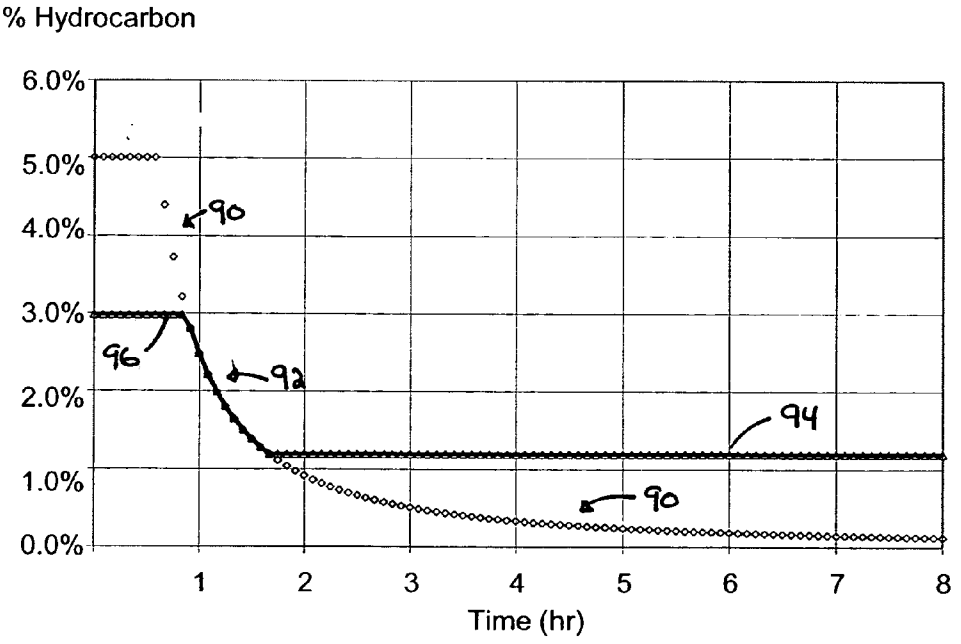


Figure 3

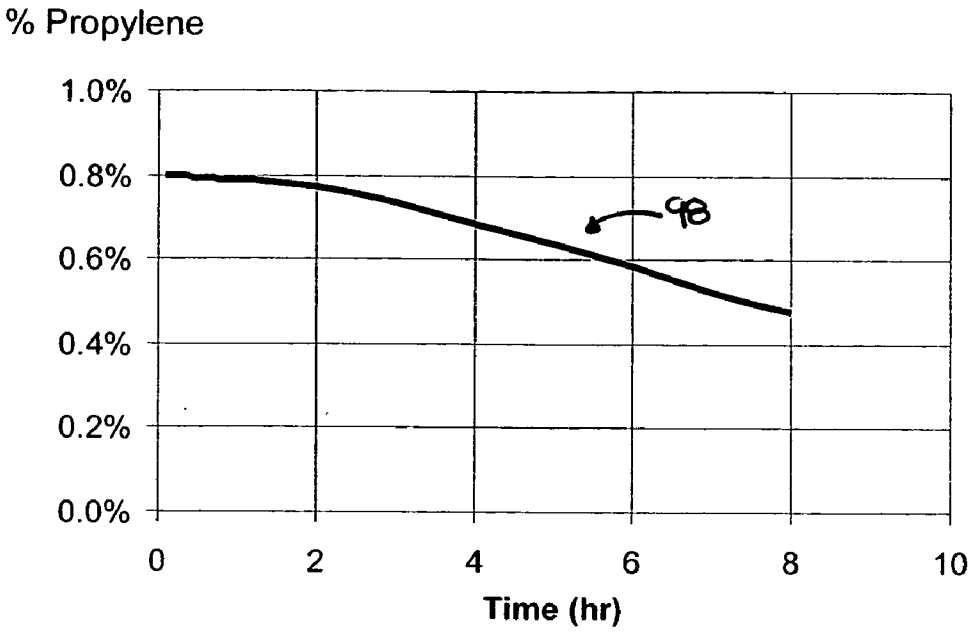


Figure 4

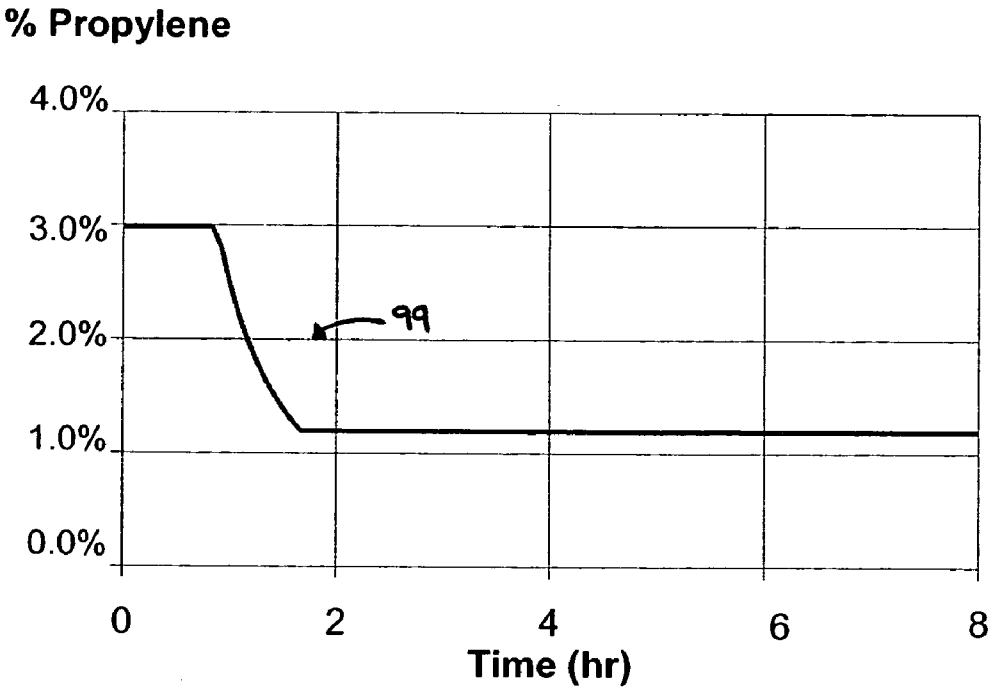


Figure 5

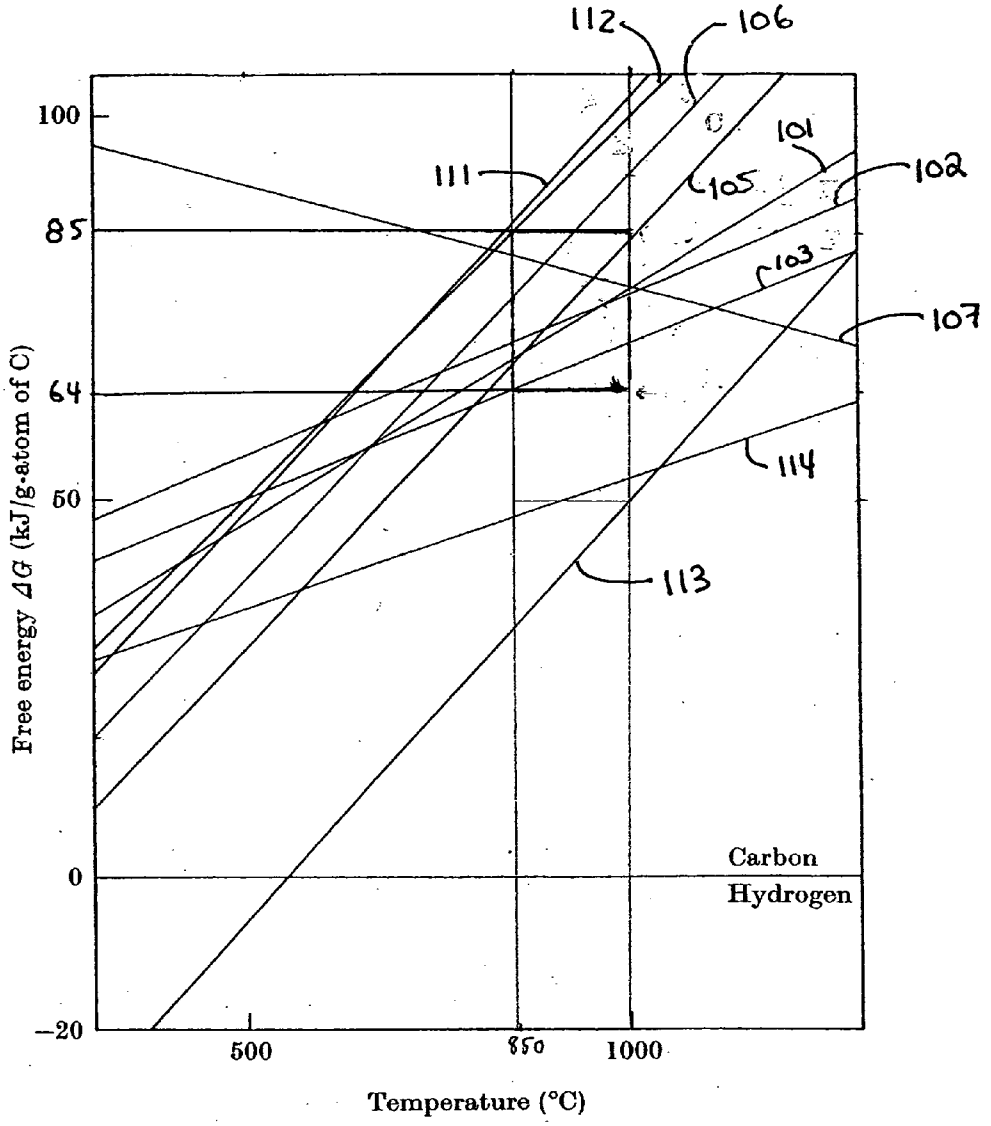

112
106
100
101
111
102
105
85
103
107
64
114
50
113
Carbon
Hydrogen
0
−20
500
850
1000
Free energy ΔG (kJ/g-atom of C)
Temperature (°C)

Figure 6

METHOD FOR OXYGEN FREE CARBURIZATION IN ATMOSPHERIC PRESSURE FURNACES

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] None

[0002] 1. Field of the Invention

[0003] The present invention relates to methods of carburizing metal parts in an oxygen-free gas atmosphere, and more particularly to a method of varying in a controlled way the hydrocarbon and hydrogen gas concentrations in the furnace atmosphere to carburize metal parts with very low levels of inter-granular oxidation, and without formation of cementite or other metallic carbides in and/or soot on the carburized metal part.

[0004] 2. Background

[0005] Carburizing is a well-known process by which carbon atoms are adsorbed on the surface of a metal part and are introduced into the metal part via diffusion and is intended to make the surface of the metal part harder and more abrasion resistant. Carburization of steel generally involves a heat treatment of the metallic surface using a gaseous or plasma source of carbon within a furnace. Current carburization practice typically includes creating an endo-gas type of atmosphere containing nitrogen, hydrogen, a hydrocarbon, and an oxygen-containing compound such as carbon monoxide.

[0006] Unfortunately, endo-gas atmospheres are somewhat difficult to generate and precisely control, are typically more costly to operate, and involve additional safety risks due to the gas atmosphere flammability and toxicity. In addition, because the endo-gas atmosphere includes oxygen-laden gases, the resulting formation of inter-granular oxidation (IGO) within the metal part cannot be avoided.

[0007] Optimization of the carburization process requires careful control of both the ambient gas composition and the furnace temperature. Clearly, temperature control of the furnace is required as the heat within the furnace impacts the microstructure of the metal part and the reaction equilibriums and kinetics. Precisely controlling the ambient gas composition is also required so as to optimize the carbon content of the ambient gas composition, as too great a concentration of carbon may make metal part brittle and unworkable. Likewise, control of the ambient gas composition within the furnace has been used in efforts to try to limit undesirable effects of carburization, namely the formation of inter-granular oxidation, cementite or other metallic carbides, and soot, as well as avoiding hydrogen embrittlement. Unfortunately, the current methods of controlling endo gas type atmospheres during the carburization process in an atmospheric pressure furnace have often resulted in a trade-off or balancing of the undesired effects in the carburized metal part and are not able to avoid IGO.

[0008] Previously, where greater control over the ambient gas composition is desired, carburization may take place under very low pressures in a vacuum chamber. Such vacuum carburization process allows for the use of less complex gas atmospheres when compared to endo-gas atmosphere systems and can produce parts with low inter-granular oxidation, but require expensive and complex vacuum furnaces. In addition, such vacuum carburization process typically operate in a very narrow range of low pressure, as too low of a pressure yields inadequate or inefficient carburization rates, and too high a pressure (above about 0.1 atmosphere) produces undesirable sooting of the metal parts. Also, the low atmosphere density within a vacuum carburization process and system makes it difficult to carburize blind holes and recesses.

[0009] What is needed, therefore, is an efficient carburization technique for use in atmospheric pressure furnaces that minimizes formation of inter-granular oxidation, and avoids undesirable effects such as hydrogen embrittlement, cementite or other carbide formation, and soot formation.

SUMMARY OF THE INVENTION

[0010] The present invention may be characterized as a method of treating a metal part in an atmospheric pressure furnace comprising the steps of: (a) introducing the metal part into the atmospheric pressure furnace having a gas atmosphere substantially free of oxygen, the gas atmosphere comprising a carbon containing gas substantially free of oxygen, such as a hydrocarbon gas and a reducing gas, such as hydrogen; (b) heating the metal part to a target temperature; (c) adjusting the gas concentrations of the gas atmosphere to inhibit formation of metal oxides in the metal part at the target temperature and to maintain a carbon saturation factor of between about 0.75 and 1.00 with negligible cementite formation; and (d) removing the metal part from the atmospheric pressure furnace.

[0011] In another aspect, the present invention may be characterized as a method of treating a metal part comprising the steps of: (a) preparing the atmospheric pressure furnace with a controlled gas atmosphere substantially free of oxygen and including hydrogen gas; (b) adjusting the concentration of the hydrogen gas above a prescribed minimum threshold to inhibit formation of metal-oxides at a target temperature; (c) introducing a hydrocarbon gas into the controlled gas atmosphere; and (d) treating a metal part in the controlled gas atmosphere of the atmospheric pressure furnace at the target temperature.

[0012] In yet another aspect, the present invention may be characterized as a method of treating a metal part comprising the steps of: (a) preparing the atmospheric pressure furnace with a controlled gas atmosphere substantially free of oxygen and including a hydrocarbon gas; (b) heating the metal part at a target temperature in the controlled gas atmosphere of the atmospheric pressure furnace for a prescribed duration; and (c) adjusting the concentration of the hydrocarbon gas as a function of time to maintain a carbon saturation factor of between about 0.75 and 1.00 with negligible cementite formation throughout the prescribed duration.

BRIEF DESCRIPTION OF THE DRAWINGS

[0013] The above and other aspects, features, and advantages of the present invention will be more apparent from the following, more detailed description thereof, presented in conjunction with the following drawings, wherein:

[0014] FIG. **1** is a schematic representation of the oxygen-free carburization system in accordance with the present invention;

[0015] FIG. **2** is a graph that depicts the amount of oxygen required for the formation of chromium oxide compared to the equilibrium concentration of oxygen in the furnace atmosphere as a function of added hydrogen at about 900 degrees Centigrade;

[0016] FIG. **3**, FIG. **4**, and FIG. **5** depict various hydrocarbon gas concentrations as a function of time in accordance with embodiments of the present invention; and

[0017] FIG. **6** is a graph depicting the free energy of formation for selected hydrocarbons.

DETAILED DESCRIPTION

[0018] Referring now to FIG. **1**, there is shown a schematic representation of the present oxygen-free carburization control system **10**. The oxygen-free carburization control system **10** employs a gas control panel **12** to control the flow and mixing of a specific gas mixture and deliver that specific gas to an atmospheric pressure furnace **20**. For purposes of this document atmospheric pressure furnace broadly means any non-vacuum furnace and more particularly means furnaces operating at pressures between 10 psig and slightly below atmospheric.

[0019] The controlled gas atmosphere includes a variable combination of: a reducing gas; and a carbon containing gas with the balance being an inert gas, all such constituent gases being substantially free of oxygen. In the preferred embodiment, the specific gas mixture **60** includes a variable combination of nitrogen gas **14**, hydrogen gas **16**, and hydrocarbon gas **18** in prescribed concentrations and is delivered to the atmospheric pressure furnace **20** as a function of time. The gas atmosphere within the furnace is thus substantially oxygen free, with very small quantities of oxygen present as a result of leakage, impurities, etc.

[0020] A series of user inputs **22** defining the desired process parameters are collected via the control panel **12**. The desired process parameters help characterize the final carbon profile desired in the metal part to be carburized, and would preferably include the desired case depth, desired carburization temperature, initial carbon content, target saturation factor, maximum inter-granular oxidation (IGO) allowable, etc. Other user inputs **22** may include results or analysis from test samples from the furnace during the calibration and operation of the furnace. Preferably such results or analysis would include actual carbon uptake realized in the furnace for given steel alloys at given atmosphere conditions. In addition, a series of sensed or measured parameters including furnace temperature **24** as measure with a temperature sensor, and furnace oxygen content **26** as measured with an oxygen probe, are also preferably input to the gas control panel **12**.

[0021] The control panel **12** then uses a carburization model **30** to calculate a profile of nitrogen gas **14**, hydrogen gas **16**, and hydrocarbon gas **18** concentrations as a function of time. The model **30** is preferably a software routine that uses selected inputs, including user inputs **22**, furnace temperature **24**, furnace oxygen content **26**, as well as known parameters such as alloy composition, furnace type, etc. to calculate or ascertain the desired gas concentrations as a function of heat treatment time.

[0022] Metal parts **70** to be carburized are loaded into an atmospheric pressure furnace **20** and contacted with the prescribed gas mixtures **60** for the prescribed durations. Once heat treated, the treated metal parts **72** are removed from the atmospheric pressure furnace **20** and placed in a cooling or quench chamber **40**. The cooling or quench chamber **40** also comprises an atmosphere that is substantially free of oxygen so as to further minimize oxidation. The carburized metal part **72** is cooled or quenched for a prescribed duration under controlled conditions after which the heat-treated metal part **74** is removed. The gas atmosphere can be released or exhausted via a vent **42**, as appropriate.

[0023] Flow of the nitrogen gas **14**, hydrogen gas **16** and hydrocarbon gas **18** are controlled to the atmospheric pressure furnace **20** using control valves **34**, **36**, and **38** which are controlled or adjusted by the gas control panel **12** in response to the model **30** and feedback or inputs from the corresponding flow meters **44**, **46**, and **48**. The individually metered gas streams **54**, **56**, and **58** are then mixed and the resulting gas mixture **60** is introduced to the atmospheric pressure furnace **20** in accordance with the model **30**.

[0024] The preferred oxygen-free heat treatment or carburization process involves the following typical sequence of steps: (1) Seasoning the atmospheric pressure furnace with a nitrogen, hydrogen, and hydrocarbon gas atmosphere to remove or reduce the oxygen and moisture content within the furnace and to equilibrate the furnace materials with the carburizing atmosphere; (2) Calibrating the carburization recipe or model for the nitrogen, hydrogen, and hydrocarbon gas atmosphere; (3) Establishing user inputs including case depth, treatment temperature, initial carbon content, saturation factor, etc.; (4) Generating an initial profile of desired hydrocarbon gas in the furnace as a function of time; (5) Changing the furnace atmosphere to a substantially nitrogen and hydrogen gas atmosphere; (6) Loading the metal parts into the furnace atmosphere until such metal parts achieve the desired treatment or process temperature; (7) Adjusting the hydrocarbon gas concentration, hydrogen gas concentration, and nitrogen gas concentration in the furnace in accordance with the model or recipe; (8) Removing the treated metal parts when the treatment cycle is complete and placing the treated metal parts into a quench or cooling chamber that, preferably, is substantially free of oxygen; and (9) Cooling (e.g. quenching) the treated metal parts in the substantially oxygen free atmosphere.

[0025] Seasoning of a furnace is characterized as a process that uses an inert gas plus a carbon source to purge or reduce the oxygen and moisture content within the furnace as well as to eliminate or reduce the carbon uptake variability within the furnace prior to the start of the heat treating process. In the preferred embodiment, the seasoning atmosphere within the furnace includes a small concentration of hydrocarbon gas (e.g. approximately 0.5% to 2.0% propylene gas), about 5.0% hydrogen gas, and the balance of the introduced gas atmosphere being nitrogen gas. The seasoning step should proceed for a time sufficient to allow the furnace to equilibrate at the desired process or treatment temperature. This is usually determined by steady measurements from an oxygen probe disposed within the furnace at the desired process or treatment temperature.

[0026] Calibration of the oxygen-free carburization model for the nitrogen, hydrogen, and hydrocarbon gas atmosphere is preferably accomplished using one or more test samples or shims. The test sample or shim is introduced into the seasoned furnace atmosphere comprising constant volumes of nitrogen gas, hydrogen gas a small percentage of hydrocarbon gas. The shim remains in the furnace atmosphere for about 30 minutes or a time sufficient to soak at the desired process or treatment temperature. The test sample or shim is then removed from the furnace and the actual carbon uptake is determined and with other user inputs entered into the oxygen-free carburization model or recipe.

[0027] After calibration of the oxygen-free carburization model is complete, user inputs are received and an initial profile of the ambient gas atmosphere of the furnace as a function of time is produced. Prior to the actual heat treating process of the metal parts, the hydrocarbon gas flow is briefly interrupted and the furnace atmosphere is changed to a nitro-

gen and hydrogen gas atmosphere. The metal parts are loaded into the nitrogen and hydrogen gas atmosphere and heated until such parts attain the desired treatment or process temperature. Upon attaining the desired treatment or process temperature, adjustment of the hydrogen gas, hydrocarbon gas, and nitrogen gas concentrations in the furnace atmosphere is initiated.

[0028] As explained in more detail below, the step of adjusting the hydrocarbon gas concentration in accordance with the model typically involves varying the hydrocarbon gas flow to achieve a desired concentration of hydrocarbon gas in the furnace atmosphere as a function of time. The desired range of hydrocarbon gas concentration is from about nil or 0.0% to about 10.0% and more preferably in the range of between about 0.3% to about 5.0%. The actual hydrocarbon concentration is calculated using a carbon saturation factor within the process model.

[0029] A minimum concentration of hydrocarbon gas in the furnace atmosphere has been found to be preferred in order to achieve reasonable levels of carbon flux on the metal parts to be treated. It will be understood that some of the hydrocarbon gas is consumed through reacting with oxidizing species that are continuously introduced into the furnace through leaks, door openings, etc, and this can vary from one furnace and implementation to another. Upon initiation of hydrocarbon gas flow, the preferred minimum concentration of hydrocarbon gas within the furnace is likely in the range of 0.1% to 2.0%, and more preferably in the range of 0.3% to 1.5%. This minimum level of hydrocarbon gas within the furnace atmosphere is easily determined by shim tests as the present model is calibrated for a given furnace.

[0030] Similarly, a maximum hydrocarbon gas concentration within the furnace atmosphere may be set due to practical flow rate limitations of the control valves and lines employed as well as economic or safety considerations. Maximum hydrocarbon concentrations in the furnace atmosphere are typically in the range of about 4.0% to about 10.0%. For optimum process performance, the preferred hydrocarbon is propylene and the gas concentration range of the propylene is preferably between about 1.0% to about 3.5% concentration of the substantially oxygen-free gas atmosphere in the furnace.

[0031] The step of adjusting the hydrogen gas concentration in the furnace in accordance with the model or recipe comprises setting the hydrogen gas flow to achieve a desired concentration of hydrogen gas in the furnace atmosphere. The desired concentration of hydrogen gas is calculated within the model and is preferably between a prescribed threshold minimum concentration and a prescribed threshold maximum concentration. As explained in more detail below, the minimum concentration of hydrogen gas depends on several factors including the alloy elements within the metal part to be treated with potential for oxide formation at the process or treatment temperature; and the quantity of oxygen, moisture, and other impurities present in the furnace. The maximum concentration of hydrogen gas is preferably determined based on consideration of several other process factors, such as flammability of the gas, potential for decarburization, potential for hydrogen embrittlement, and hydrogen gas cost. In the described embodiment, the preferred range of hydrogen gas is between about 1.0% to about 10.0% and more preferably in the range of between about 1.5% to about 5.25% concentration of hydrogen gas in the furnace.

[0032] The final step of cooling (e.g. quenching or tempering) the carburized metal parts preferably in a substantially oxygen free atmosphere also helps to minimize the formation of inter-granular oxidation. Upon completion of the quenching step, the quenching atmosphere is vented and the heat treatment process is complete. It should be noted that the quenching process may utilize various quenching techniques including intensive quenching with water or a cryogen or standard quenching with oil. Alternatively, the cooling may be done more gradually at ambient temperatures in a nitrogen atmosphere or other oxygen free atmosphere for a prescribed period of time.

Calculation of Hydrogen Gas Concentration

[0033] For certain steel alloys, particularly those containing Cr, Ti, Mn, and/or Si, the presence of oxygen in the gas atmosphere within the furnace promotes the formation of inter-granular oxidation (IGO) within the treated part. Introduction of hydrogen gas in sufficient concentrations into the gas atmosphere within the furnace negates the formation of the likely metal oxides in favor of reactions between hydrogen and oxygen. The actual concentration of hydrogen gas necessary to reduce or negate the formation of inter-granular oxidation (IGO) is ascertained or calculated based on the thermodynamic equilibrium between the potential oxidized products and reactants within the system. The oxidation potential observed with hydrogen varies based on the concentration of hydrogen gas in the furnace. The reactants can include not only hydrogen, water, and oxygen, but also include species containing carbon, such as hydrocarbons, carbon monoxide, and carbon dioxide. For simplicity, the procedure described herein is limited to reactants including hydrogen, water, and oxygen.

[0034] Oxidation of the metals of interest, including Cr, Ti, Mn, and/or Si, are well known and documented processes that depend, in part, on given furnace temperatures and operating conditions. The partial pressure of oxygen (pO2) in equilibrium with the metal oxide is given by the following equation:

$$pO_2=\exp(\Delta G^o/RT);$$

[0035] where 'T' is the furnace temperature in Kelvin (K), 'R' is the gas constant (=8.3 J/(mole*K) and ΔG^o is the Gibbs free energy (J/mole) for the reaction a $Me+O_2>>>MeaO_2$ (assuming ideal behavior and pure metal).The partial pressure of oxygen available in the furnace atmosphere is calculated based on the equilibrium relationship between hydrogen, oxyen and water $2H_2O=2\ H_2+O_2$.

[0036] A comparison is made between the partial pressure of oxygen required to form oxides of the metals of interest and the equilibrium concentration of oxygen in the furnace atmosphere with varying amounts of hydrogen gas in a given furnace under prescribed conditions. Included in these comparisons and calculations are the quantity of O_2 and moisture (i.e. H_2O) initially present in the seasoned furnace atmosphere as adjusted to account for additional concentrations of O_2 and H_2O attributable to impurities subsequently introduced in the furnace. The adjusted quantity of O_2 and moisture (i.e. H_2O) in the furnace at the given operating conditions is preferably estimated from past experience with the furnace or determined through experimentation or calibration testing of the furnace.

[0037] The minimum concentration of hydrogen gas to be used in the ambient gas atmosphere in the furnace is the calculated amount of hydrogen that results in a lower partial

pressure of oxygen than is required to form oxides of the metals of interest found in the alloy of the part to be carburized. It should be understood that the minimum amount of hydrogen gas needed to limit inter-granular oxidation (IGO) formation may vary depending not only on the gas composition, but also physical construction and operation of the furnace and the actual composition of the steel alloy. For example, a furnace that allows a greater flow rate of outside atmosphere containing oxygen and/or moisture into the furnace would require a higher concentration of hydrogen gas to achieve the desired low value of oxygen partial pressure (pO2). From a practical standpoint, minimizing inter-granular oxidation (IGO) to 0.0007 inches or below meets the SAE Aerospace Material Specification 2759/1C for heat treatment of carbon and low-alloy steel parts.

[0038] On the other hand, the maximum concentration of hydrogen gas used in the ambient gas atmosphere in the furnace is determined based on consideration of several other factors, such as flammability of the gas, potential for decarburization, potential for hydrogen embrittlement, and economic impacts of such hydrogen gas use. It has been shown that hydrogen gas concentrations within a carburizing furnace up to about 40% can increase the carburization rate of hydrocarbons in nitrogen by several fold. However, when using the present model and oxygen-free carburization process, it has been found that the increase in carburization rate lessens above hydrogen gas concentrations of about 10%. Also, at hydrogen concentrations substantially above 10%, other adverse effects become apparent including hydrogen embrittlement of the metal part. Limiting the hydrogen gas concentration to a maximum of about 10% avoids such adverse effects.

[0039] From a safety standpoint, limiting the hydrogen gas concentration within the furnace is also advisable. The National Fire Protection Association (NFPA) has set a 10% hydrogen gas concentration in ambient atmosphere conditions as constituting an explosive concentration, which makes 10% hydrogen gas concentration a reasonable maximum concentration for safety reasons. Still further, the lower explosive limit (LEL) for hydrogen gas in air at ambient conditions is about 4.0%, and the minimum oxygen concentration required for hydrogen flammability is about 5.0%. Thus, if the oxygen-free carburizing atmosphere within the furnace contains a hydrogen gas concentration of 5.25% or less, then no mixture of furnace atmosphere and ambient air (from outside the furnace) would produce both a hydrogen gas concentration and oxygen concentration above the minimums required for flammability. Therefore, from a safety standpoint, a more preferable range of hydrogen gas concentration is between about 1.5% and about 5.25%.

[0040] FIG. **2** depicts a graph that compares the partial pressure of oxygen that is required to form chromium oxide in a gas atmosphere furnace to the partial pressure of oxygen available in the furnace as a function of added hydrogen. As seen therein, the partial pressure of oxygen required to form the oxide of pure chromium (Cr) at 900 degrees C. is about 6.0×10^{-25} and is constant as the concentration of hydrogen gas within the furnace varies. Conversely, the partial pressure of oxygen available in the furnace as a function of hydrogen (H_2) at 900 degrees C. ranges from about 2.59×10^{-23} at about a 0.4% concentration of hydrogen gas within the furnace to about 4.14×10^{-26} at about a 10% concentration of hydrogen gas. In this example, the gas atmosphere of the seasoned furnace is estimated to have about 1 parts per million (ppm) O_2 and about 1 ppm of H_2O, as adjusted for impurities. Using the graph in FIG. **2** and assuming the metal part to be carburized includes significant amounts of chromium and no significant amounts of other metals of interest having high oxidation potentials, the amount of hydrogen concentration in the furnace atmosphere required to substantially prevent oxidation of chromium in the metal part is about 3% or more concentration of hydrogen gas.

[0041] Steel alloy compositions of the most prevalent forms of steel alloy are presented in Table 1 below. As seen therein, chromium is often the element of interest with oxidation potential in the Steel alloys SAE 8620, SAE 9310, and SAE 4140 at the typical carburization temperatures (1700 degrees F.).

TABLE 1

Steel Alloy Element Compositions

Element	SAE 1010	SAE 4140	SAE 4820	SAE 9310	SAE 8620
C (%)	0.08–0.13	0.38–0.43	0.18–0.23	0.08–0.13	0.18–0.23
Mn (%)	0.30–0.60	0.75–1.00	0.50–0.70	0.45–0.65	0.70–0.90
P_{max} (%)	0.04	0.035	0.035	0.035	0.035
S_{max} (%)	0.05	0.040	0.04	0.04	0.04
Si (%)	0.15–0.35	0.15–0.30	0.15–0.35	0.15–0.35	0.15–0.35
Ni (%)	. . .	. . .	3.25–3.75	3.00–3.50	0.40–0.70
Cr (%)	. . .	0.80–1.10	. . .	1.00–1.40	0.40–0.60
Mo (%)	. . .	0.15–0.25	0.20–0.30	0.08–0.15	0.15–0.25

Calculation of Hydrocarbon Gas Concentration

[0042] The primary objective of varying the concentration of hydrocarbon gas in the furnace is to maintain a desired net carbon flux from the furnace atmosphere that is less than the carbon flux required to saturate the surface of the metal part with carbon. By "saturate," we mean that the carbon concentration in the metal is at a specified level. For example, the specified level may be the maximum carbon concentration at a given temperature that would avoid formation of a certain phase, such as cementite or other metallic carbide. Alternately, the specified level may be a desired surface carbon concentration, for example the starting bulk carbon concentration in the case of a neutral hardening heat treatment. The specified level denoting "saturation" may change depending on composition of the metal, temperature, or time in the heat treating process. Moreover, as carbon transport occurs across the gas and solid interface, it is desirable for the net carbon flux from the gas flow to the surface of the metal part and carbon diffusion of atoms within the metal part to match or approximate one another. Adjusting the concentration of hydrocarbon gas within the furnace allows control of the carburization process such that: (1) the carbon flux from the atmosphere gas flow to the metal surface matches or approximates the diffusion of carbon atoms within the metal part; and (2) the carbon flux from the atmosphere gas flow to the metal part is less than the carbon flux that would saturate the surface of the metal part with carbon. Controlling both aspects results in avoidance or minimization of soot and cementite formation (or other undesirable carbide) at or near the surface of the treated metal part.

[0043] As the oxygen-free carburization process continues, both the carbon flux required to saturate the surface of the metal part with carbon as well as the carbon diffusion of atoms within the metal part change as a function of time.

Accordingly, the concentration of hydrocarbon gas within the furnace should likewise be adjusted as a function of time.

[0044] In the present embodiment, the desired hydrocarbon gas concentration is determined based on the actual measured carbon flux data from the furnace together with an estimated or calculated diffusivity of carbon in the treated metal part. Measuring the carbon flux is preferably accomplished using an appropriate methods such as shim testing, resistivity measurements of a thin wire disposed within the furnace, or other methods known to those skilled in the art and used to determine rate of carburization or carbon content of the metal parts being treated at known operating conditions.

[0045] The calculated diffusivity of carbon or carbon diffusion profile in the metal part is calculated based on Fick's second law, presented below, which is used to determine the flux of carbon with time into a metal part:

$$\frac{\partial C}{\partial t} = D\frac{\partial^2 C}{\partial x^2}$$

[0046] where 'C' is the carbon atom concentration of the part, 'x' is the distance from the surface of the part that carbon atoms must diffuse into the part, 'D' is the diffusion coefficient of carbon atoms into the metal, and 't' is time. As is well known in the art, the diffusion coefficient generally increases exponentially with the reciprocal of the temperature.

[0047] The calculation of the carbon diffusion profile in the metal can be performed by assuming that a carbon source is supplied directly to the metal surface, as is the case in the carburization furnace, and the metal part is treated as a semi-infinite slab. The approximated solution of Fick's second law thus becomes:

$$(C_s-C_x)/(C_s-C_o)=\text{erf}\{x/2(Dt)^{1/2}]\}$$

[0048] where C_s is the carbon concentration at the surface, C_o is the original carbon concentration in the metal, and C_x is the carbon concentration at the distance x, from the surface of the metal part into the body of the metal part, and the value 'erf $\{x/[2(Dt)^{1/2}]\}$' is the Gaussian Error Function. Note, the carbon concentration at the surface, C_s, is the specified level or saturation concentration. Using this estimate of diffusive carbon flux, it is possible to calculate the carbon profiles at any time during the treatment process and used as a benchmark for process control.

[0049] The carbon concentration at the surface of the metal part is determined empirically by taking carbon flux measurements of samples in known furnace atmosphere conditions. The carbon flux measurements are used to determine the amount of carbon (i.e. hydrocarbon gas) that is delivered to the surface of the metal part over time, at different atmosphere compositions, and different furnace temperatures. Carbon flux measurements are taken with foil samples of known surface area that are exposed to a known atmosphere compositions.

[0050] A carbon saturation factor Φ, is then applied to assure that the amount of carbon delivered to the surface of the part is below that which would produce cementite, other metallic carbides, soot or, in the case of neutral hardening for example, would prevent decarburization on the surface of the metal part. The carbon saturation factor Φ, is represented by the equation:

$$\Phi = \frac{(Flux_{Fuel})}{(Flux_{Metal})}$$

[0051] where Flux_{Fuel} is the carbon flux rate from the hydrocarbon gas to the surface of the metal part and is determined empirically from the measurements taken from the samples exposed to the known atmosphere compositions; and Flux_{Metal} is the sum of the calculated diffusive flux of carbon away from the surface of the metal part taken from the above carbon diffusivity estimate, and the carbon flux leaving the surface due to any decarburizing effect of reactions with the gas atmosphere. In carburizing applications the diffusive flux is usually the critical component of the flux away from the metal surface. In neutral hardening applications, the diffusive flux is usually negligible due to a uniform carbon concentration throughout the metal, such that the decarburizing flux is the critical component of the flux away from the metal surface. In applications where the decarburizing flux is significant, it can be either calculated by methods known in the art based on estimates or measurements of decarburizing specie concentration in the gas atmosphere, or determined empirically by test.

[0052] The theoretical ideal carbon saturation factor, Φ, is between about 0.75 and 0.99. However, given variation in accuracy of sample measurements and in precision of flow and temperature control, a practical range for the saturation factor is preferably between about 0.75 and 1.25, and more preferably between about 0.90 and 1.10. Using the preferred saturation factor range and the Flux_{Metal} value calculated from the above diffusivity model, an optimal or target value of Flux_{Fuel} is determined. The preferred hydrocarbon gas concentration as a function of time is then determined based on the optimal or target value of Flux_{Fuel} so that the carbon saturation factor Φ, is maintained in the desired range during any time interval of the process treatment. The relationship between Flux_{Fuel} and hydrocarbon gas concentration is determined empirically and periodically updated using actual foil sample data from the actual furnace.

[0053] The preferred hydrocarbons useful with the present embodiments of the invention include those high purity hydrocarbons having intermediate stability at typical carburization temperatures (e.g. 1550 to 1700 degrees F.). Hydrocarbons of intermediate stability can be selected based the value of free energy of formation per gram-atom of carbon.

[0054] As shown in FIG. **6**, hydrocarbons with a free energy of formation per gram-mole carbon between about 64 and 85 kJ/gmol over the carburization temperature range of 850-1000° C. provide the best balance of carburization rate and avoidance of soot or carbide formation. This includes hydrocarbons such as propylene **101**, butadiene **102**, ethylene **103**, butene (not shown), ethane **105**, propane **106**, and acetylene **107**. Also shown in FIG. **6**, are hexane **111**, Cyclohexane **112**, methane **113** and benzene **114**. Even more preferred sources of hydrocarbon for use with the present methods include unsaturated hydrocarbons and propylene is the most preferred hydrocarbon. The molecular orbitals of carbon-carbon double bonds found in unsaturated hydrocarbons are known to interact with the molecular orbitals of transition metals such as iron. In the proposed process, this effect promotes the pyrolysis reaction at the surface of the metal parts as opposed to reactions in the bulk gas phase. Such promotion of reaction at the metal surface helps to minimize soot for-

mation and improve process efficiency and control. In the case of propylene, an important reaction pathway for pyrolysis involves the breakdown of the propylene molecule into methane and acetylene. The thermodynamic stability of the methane byproduct makes this pathway kinetically favored, and the instability of acetylene causes a fast subsequent breakdown to carbon at the surface, resulting in favorable carburization rates for propylene. The higher stability of propylene in the gas phase as compared with acetylene serves to minimize soot formation and improve control.

[0055] Application of the above-described diffusive carbon flux model to the present oxygen-free carburization process is preferably accomplished in the following manner: (1) Ascertain the known parameters C_s, C_o, D, D_o, R, T and Q from the properties of the metal part and carburization process targets and/or constraints; (2) Specify the desired final carbon profile or case depth, x; (3) Use the model to calculate the target carbon flux as a function of time, t, or the target carbon concentration at the surface of the metal part, C_s as a function of time, t; and (4) Establishing the hydrocarbon gas content that achieves the desired carbon concentration at the surface of the metal part, C_s using the actual measured carbon flux data from the shim tests or other test methods together with the user defined saturation factor Φ; (5) Set the furnace processing conditions to achieve the hydrocarbon gas concentration as a function of time. Upon completion and execution of the carbon diffusivity model, the oxygen-free carburization process, as set forth above is continued. The final result of the oxygen-free carburization process performed in accordance with the model should achieve the desired case depth and carbon profile in the treated metal part with little or no intergranular oxidation (IGO), soot formation or cementite or other metallic carbide formation. Some testing and adjustment of operating conditions may be used to adapt the model and associated procedures to the conditions and variability of a particular furnace, material, surface finish, etc.

[0056] An optimum hydrocarbon gas concentration profile may involve three or four phases including: (a) an initial high hydrocarbon concentration phase; (b) a moderate hydrocarbon concentration phase; (c) a lower hydrocarbon concentration phase; and (d) zero or nil hydrocarbon concentration phase. For carburizing applications, the most preferred profile would be a continuously varying hydrocarbon concentration, beginning with the high concentration phase and ending with the low or zero concentration phase. This would provide the closest match to the diffusive flux and minimize carburization time.

[0057] The initial high hydrocarbon phase is intended or adapted to bring the carbon content at the surface of the part rapidly up to at or near the constraint level (i.e. 1.2% C, or as high as practical so as not to form carbides on the surface of the metal part). The moderate hydrocarbon concentration phase is intended or adapted to maintain the carbon content at the surface at the desired percent (e.g. about 1.2% C) while the carbon atoms diffuse into the body of the metal part. Put another way, the moderate hydrocarbon concentration phase attempts to match the rate of carbon delivery to the surface of the metal part to the rate of carbon diffusion into the body of the metal part. The lower hydrocarbon concentration phase is intended or adapted to allow the carbon content at the surface of the metal part to decline to a lower level, for example where the process conditions or carburization requirements call for a surface carbon content of less than 1.2% C. Finally, the zero or nil hydrocarbon concentration phase is intended or adapted to allow the carbon profile to reach the desired end state. This zero or nil phase may occur near the end of the carburization process to remove any trace of free carbon from the surface of the treated metal part or may also be used during the cooling or quenching operations.

[0058] A minimum concentration of hydrocarbon gas in the furnace atmosphere has been found to be needed to achieve reasonable rates of carbon flux on the metal parts to be treated. Some of the hydrocarbon gas feed is consumed by reacting with oxidizing species that are continuously introduced into the furnace through leaks, door openings. This minimum concentration of hydrocarbon gas varies depending on rates of impurities in the furnace, but is likely in the range of 0.1% to 2.0%, and more probably in the range of 0.5% to 1.5%. The level is easily determined by shim tests when the model is calibrated for a given furnace or with an oxygen probe.

[0059] A maximum hydrocarbon concentration may be set due to practical flow rate limitations of the hardware utilized, economic considerations, or more preferably safety considerations. For example, the lower explosive limit (LEL) for propylene in air is about 2.4%, and the minimum oxygen concentration required for flammability is about 5.0%. If the carburizing atmosphere contains propylene at about 3.15% or less, then no mixture of furnace atmosphere and ambient air (i.e. outside the furnace) would produce a propylene concentration above the LEL coincident with an oxygen concentration above the minimum required for flammability. Therefore from a safety aspect, the preferred embodiment would set a maximum propylene concentration of 3.15% to ensure a non-flammable atmosphere during operation. Broadly speaking, hydrocarbon concentrations in the range of 0% to about 10% are recommended, more preferably in the range of about 1% to about 5%.

[0060] FIG. **3** shows a graph depicting the model hydrocarbon concentrations **90** as a function of time based on the outputs of the model compared with the actual hydrocarbon concentrations **92** employing the practical or prescribed threshold minimum concentrations **94** and maximum concentrations **96** as discussed herein.

EXAMPLES

Example 1

[0061] This example involved the carburization of a piece of ¾"×2" SAE 9310 steel alloy rod to a desired effective case depth of about 0.050 inches using the oxygen-free carburization model. A nitrogen atmosphere containing 0.6% propylene and 5.0% hydrogen was equilibrated in a laboratory tube furnace atmosphere at about 1700 degrees F. until the O_2 concentration stabilized. The carbon uptake in the tube furnace was empirically determined to be about 1.0% based on a SAE 1010 shim sample (0.001" thick) placed in the furnace and contacted with the oxygen-free gas atmosphere for about 15 minutes.

[0062] The relevant process inputs were entered into the oxygen-free carburization model and a desired hydrocarbon gas concentration profile as a function of time was established to achieve the desired effective case depth. The hydrocarbon gas concentration profile **98** used is depicted in FIG. **4**. Hydrogen gas concentration during the carburization process was maintained at about 5.0%. The sample was placed in a loading vestibule and purged with nitrogen to remove residual oxygen. The sample was then introduced to the furnace atmosphere at about 1700 degrees F. and allowed to come to temperature. The propylene gas flow was then introduced according to the predicted hydrocarbon gas profile **98**.

[0063] After about 8 hours, the hydrogen and propylene were turned off, the sample was removed from the furnace and water quenched. Analysis of the sample showed an effective case depth of about 0.058 inches and there was no visible sooting (see Table 2). Trace amounts of inter-granular oxidation (IGO) was measured at one of four points. IGO was not detected in the other three measured points. These results compare beneficially with the expected IGO level of about 0.0006 inches in a conventional endo-gas atmosphere and a case depth of 0.050 inches.

Example 2

[0064] This example involved the carburization of a SAE 4140 steel alloy sample to a desired effective case depth of about 0.047 inches in an operating atmosphere pressure carburization furnace using the oxygen-free carburization model. A nitrogen, propylene and hydrogen gas atmosphere was equilibrated in an operating atmosphere pressure carburization furnace at about 1700 degrees F. until the O_2 concentration stabilized.

[0065] The relevant process inputs were entered into the oxygen-free carburization model and a desired hydrocarbon gas concentration profile as a function of time was established to achieve the desired effective case depth. The upper hydrocarbon limit was constrained by the hydrocarbon injection equipment. The hydrocarbon gas concentration profile **99** used is depicted in FIG. **5**. Hydrogen gas concentration during the carburization process was maintained between about 1.19% and about 2.99%. The sample was placed in a loading vestibule and purged with nitrogen to remove residual oxygen. The sample was then introduced to the furnace atmosphere at about 1700 degrees F. and allowed to come to temperature. The propylene gas flow was then introduced according to the predicted hydrocarbon gas profile **99**.

[0066] After about 8 hours, the hydrogen gas and propylene gas were turned off, the sample was removed from the furnace and cooled. Analysis of the sample showed an effective case depth of about 0.048 inches and there was no visible sooting (see Table 2). Trace amounts of inter-granular oxidation (IGO) was measured and was less than or equal to 0.0002 inches. These results compare beneficially with the expected IGO level of about 0.0006 inches in a conventional endo-gas atmosphere and a case depth of 0.050 inches.

Examples 3 and 4

[0067] These examples involved the carburization of a SAE 9310 steel alloy sample (Example 3) and a SAE 4820 steel alloy sample (Example 4) to desired effective case depths of about 0.034 inches in an operating atmosphere pressure carburization furnace using the oxygen-free carburization environment but keeping the hydrocarbon gas profile constant as a function of time. A nitrogen atmosphere containing about 1.7% propylene and 5.0% hydrogen was equilibrated in an operating atmosphere pressure carburization furnace at about 1700 degrees F. until the O_2 concentration stabilized.

[0068] As with the earlier described examples, the samples were placed in a loading vestibule and purged with nitrogen to remove residual oxygen. The samples were then introduced to the furnace atmosphere at about 1700 degrees F. and allowed to come to temperature. The propylene gas flow and hydrogen gas flows were then introduced into the furnace. The hydrocarbon gas concentration profile was kept constant at about 1.7% throughout the 4 hour oxygen-free carburization treatment. Hydrogen gas concentration during the carburization process was also maintained at a constant concentration of about 5.0%.

[0069] After about 4 hours, the hydrogen and propylene were turned off, the samples were removed from the furnace and quenched. Analysis of the sample in Example 3 showed an effective case depth of about 0.034 inches, no visible sooting, and an IGO level of less than or equal to 0.0001 inches (See Table 2). Analysis of the sample in Example 4 showed an effective case depth of about 0.034 inches, no visible sooting, and no detectable level of IGO (See Table 2). These results compare beneficially with the expected IGO level of about 0.0004 inches in a conventional endo-gas atmosphere and a case depth of 0.035 inches.

Example 5

[0070] This example involved the carburizing of a SAE 1010 steel alloy sample (⅛" thick×2") to a desired effective case depth of about 0.030 inches in a laboratory tube furnace using the oxygen-free carburization model. A nitrogen atmosphere containing 0.3% ethane and 5.0% hydrogen was equilibrated in an operating atmosphere pressure carburization furnace at about 1700 degrees F. until the O_2 concentration stabilized.

[0071] The relevant process inputs were entered into the oxygen-free carburization model and a constant hydrocarbon gas concentration (e.g. about 0.3% ethane) was selected to achieve the desired treatment. Hydrogen gas concentration during the treatment process was maintained at about 5.0%. The sample was placed in a loading vestibule and purged with nitrogen to remove residual oxygen. The sample was then introduced to the furnace atmosphere at about 1700 degrees F. and allowed to come to temperature. The ethane gas flow was then introduced according to the predicted hydrocarbon gas profile.

[0072] After about 2 hours, the hydrogen gas and ethane gas were turned off and the sample was removed from the furnace and quenched. Analysis of the sample showed an effective case depth of about 0.030" and there was no visible sooting (see Table 2). Because the sample does not contain appreciable levels of alloying elements known to promote IGO, the IGO level was not measured.

TABLE 2

Oxy-Free Carburizing Test Results

	Steel Alloy	HC (type)	HC (%)	H_2 (%)	Time (hrs)	Temp (F.)	Case-Depth (in)	IGO (in)	Sooting
Ex. 1	SAE 9310	Propylene	0.44–0.80	5.0	8	1700	0.058	≦0.0003	None
Ex. 2	SAE 4140	Propylene	1.19–2.99	5.0	8	1700	0.048	≦0.0002	None
Ex. 3	SAE 9310	Propylene	1.7	5.0	4	1700	0.034	None Detected	None
Ex. 4	SAE 4820	Propylene	1.7	5.0	4	1700	0.034	0.0001	None
Ex. 5	SAE 1010	Ethane	0.3	5.0	2	1700	0.030	Not Measured	None

INDUSTRIAL APPLICABILITY

[0073] As can be appreciated from the foregoing description, the presently disclosed oxygen-free carburization process uses a precisely controlled, low-oxygen, nitrogen-based, carbonaceous atmosphere in an atmosphere pressure furnace to minimize the formation of soot, unwanted carbides, and inter-granular oxides, and avoid hydrogen embrittlement. Utilization of the above-described carburization process provides a high-quality and cost-effective process for the carburization of many steel alloys that offers many features and advantages when compared to commercially available prior art carburization processes and systems.

[0074] The present carburization process employs a substantially oxygen-free furnace atmosphere with a prescribed minimum concentration of hydrogen gas to reduce oxidation potential with trace metals in most steel alloys and to minimize the formation of inter-granular oxides (IGO). Hydrogen gas is also used as the energizer to promote dissociation of hydrocarbon fuel near and on the metal surface. As indicated above, the hydrogen gas concentration is preferably kept low to avoid hydrogen embrittlement and to improve operational safety of the carburization process by eliminating the possibility of gas explosion with low oxygen and low hydrogen concentrations. In addition, the hydrocarbon concentrations within the nitrogen-based furnace atmosphere are continuously varied to match the carbon-iron diffusion flux within the treated part for optimized carburization. The required fuel concentration to provide proper carbon flux is estimated using a flux/diffusion model together with empirically derived data obtained during calibration and operation of the oxygen-free carburization process. Moreover, the disclosed oxygen-free carburization process advantageously provides a means or technique to optimize speed of carburization through control of carbon flux.

[0075] The above-identified oxygen-free carburization methods and the features associated therewith can be utilized alone or in conjunction with other heat treatment processes and variations. Moreover, each of the specific steps involved in the carburization process, described herein, and each of the inputs, elements, or variables in the preferred carburization control system are easily modified or tailored to meet the specific treatment requirements of the particular part to be carburized or the peculiar requirements of the heat treating furnace with which it is used or other operating environment restrictions.

[0076] For example, the hydrocarbon gas could comprise propylene, propane, methane or other suitable hydrocarbon. Likewise, helium or argon gas may be used as a substitute for the nitrogen gas and dissociated ammonia may be used as a source of nitrogen gas or hydrogen gas. Also, the heat treating process can be performed in any atmospheric pressure furnace including, for example, continuous type furnaces, batch furnaces, or other furnace types (e.g. rotary retort, humpback, bell, annealing, pit, etc.) and the furnace may have a single or multiple control zones.

[0077] From the foregoing, it should be appreciated that the present invention thus provides a method of oxygen-free carburization in an atmospheric pressure furnace. While the invention herein disclosed has been described by means of specific embodiments and processes associated therewith, numerous modifications and variations can be made thereto by those skilled in the art without departing from the scope of the invention as set forth in the claims or sacrificing all its material advantages. For example, similar such methods could be employed for nitrocarburizing or ferritic nitrocarburizing by including an additional source of nitrogen such as ammonia. In addition, the present process can be adapted for use with other heat treating processes such as neutral hardening, ferritizing, annealing, normalizing, spheroidizing, tempering, sintering and sinter hardening.

What is claimed is:

1. A method of treating a metal part in an atmospheric pressure furnace comprising the steps of:

introducing the metal part into the atmospheric pressure furnace having a gas atmosphere substantially free of oxygen, the gas atmosphere comprising a carbon containing gas and a reducing gas;

heating the metal part to a target temperature;

adjusting the gas concentrations of the gas atmosphere to inhibit formation of metal-oxides on the metal part and to maintain a carbon saturation factor of between about 0.75 and 1.10 with negligible cementite or other metallic carbide formation; and

removing the metal part from the atmospheric pressure furnace.

2. The method of claim **1** wherein the carbon containing gas is a hydrocarbon gas and the reducing gas is hydrogen.

3. The method of claim **2** wherein the step of adjusting the gas concentrations of the gas atmosphere further comprise varying the volume concentration of the hydrocarbon gas in the gas atmosphere as a function of time to maintain the

carbon saturation factor of between about 0.75 and 1.10 with negligible cementite or other metallic carbide formation.

4. The method of claim **3** wherein hydrocarbon gas is selected from the group consisting of: propylene, butene, butadiene, ethylene, ethane, propane, and acetylene.

5. The method of claim **4** wherein the hydrocarbon gas has a free energy of formation per gram-mole of carbon between 64 and 85 kJ over the temperature range of about 850 degrees Centigrade to about 1000 degrees Centigrade.

6. The method of claim **4** wherein the hydrocarbon gas is propylene in a volume concentration of no greater than about 3.15 percent.

7. The method of claim **2** wherein a volume concentration of the hydrocarbon gas is between about 0.1 percent and 10.0 percent.

8. The method of claim **2** wherein the treatment of the metal part is neutral hardening and the volume concentration of the hydrocarbon gas is between about 0.2 percent and 1.0 percent.

9. The method of claim **2** wherein the treatment of the metal part is carburization of the metal part and the volume concentration of the hydrocarbon gas is between about 1.0 percent and 5.0 percent.

10. The method of claim **2** wherein the volume concentration of the hydrogen gas is between about 1.0 percent and 10.0 percent.

11. The method of claim **2** wherein the volume concentration of the hydrogen gas is between about 1.5 percent and 5.25 percent.

12. The method of claim **1** further comprising the step of seasoning the atmospheric pressure furnace to reduce the oxygen and moisture content thereof.

13. The method of claim **1** further comprising the step of cooling the metal part.

14. The method of claim **13** wherein the step of cooling the metal part further comprises cooling the metal part in an atmosphere that is substantially free of oxygen.

15. A method of treating a metal part in an atmospheric pressure furnace comprising the steps of:

preparing the atmospheric pressure furnace with a controlled gas atmosphere substantially free of oxygen and including hydrogen gas;

adjusting the concentration of the hydrogen gas above a prescribed minimum threshold to inhibit formation of metal-oxides at a target temperature;

introducing a hydrocarbon gas into the controlled gas atmosphere; and

treating a metal part in the controlled gas atmosphere of the atmospheric pressure furnace at the target temperature.

16. The method of claim **15** wherein the controlled gas atmosphere includes hydrogen gas in a volume concentration between about 1.0 percent and 10.0 percent.

17. The method of claim **15** wherein the controlled gas atmosphere includes hydrogen in a volume concentration between about 1.5 percent and about 5.25 percent.

18. The method of claim **15** wherein the step of preparing the atmospheric pressure furnace with a controlled gas atmosphere further comprises seasoning the atmospheric pressure furnace to reduce the oxygen and moisture content thereof.

19. The method of claim **15** wherein the metal part contains metals selected from the group of chromium, manganese, titanium or silicon and any inter-granular oxidation of such metals occurs at a depth of less than or equal to 0.0007 inches.

20. The method of claim **15** further comprising the step of cooling or quenching the metal part in an atmosphere that is substantially free of oxygen.

21. A method of treating a metal part in an atmospheric pressure furnace comprising the steps of:

preparing the atmospheric pressure furnace with a controlled gas atmosphere substantially free of oxygen and including a hydrocarbon gas substantially free of oxygen;

heating the metal part at a target temperature in the controlled gas atmosphere of the atmospheric pressure furnace for a prescribed duration;

adjusting the concentration of the hydrocarbon gas as a function of time to maintain a carbon saturation factor of between about 0.75 and 1.10 with negligible cementite or other metallic carbide formation throughout the prescribed duration.

22. The method of claim **21** wherein the concentration of hydrocarbon gas of the controlled gas atmosphere is adjusted to a volume concentration of between about 10.0 percent and about 0.10 percent.

23. The method of claim **21** wherein the concentration of hydrocarbon gas of the controlled gas atmosphere is adjusted to a volume concentration of between about 5.0 percent and about 1.0 percent.

24. The method of claim **21** wherein the step of adjusting the concentration of the hydrocarbon gas as a function of time further comprises:

introducing an initial maximum hydrocarbon gas concentration for a first prescribed duration;

reducing the hydrocarbon gas concentration from the maximum hydrocarbon gas concentration to a minimum hydrocarbon gas concentration over a second prescribed duration; and

maintaining the minimum hydrocarbon gas concentration for a third prescribed duration.

25. The method of claim **21** wherein the hydrocarbon gas is selected from the group consisting of: propylene, butene, butadiene, ethylene, ethane, propane, and acetylene.

26. The method of claim **25** wherein the hydrocarbon gas has a free energy of formation per gram-mole of carbon between about 64 and 85 kJ over the temperature range of about 850 degrees Centigrade to about 1000 degrees Centigrade.

27. The method of claim **21** wherein the hydrocarbon gas is propylene and the propylene gas concentration is no greater than about 3.15 percent by volume.

28. The method of claim **21** wherein there is negligible soot formation on the surface of the treated metal part.

29. A method of treating a metal part in an atmospheric pressure furnace comprising the steps of:

introducing the metal part into the atmospheric pressure furnace having a gas atmosphere substantially free of oxygen, the gas atmosphere including a carbon containing gas, a reducing gas, and an inert gas;

adjusting the gas concentrations of the carbon containing gas to maintain a carbon saturation factor associated with the metal part in the atmospheric pressure furnace of between about 0.75 and about 1.25;

wherein negligible soot and cementite, or other metallic carbide, is formed on a surface of the metal part.

30. The method of claim **29** wherein the carbon saturation factor is maintained in a range of between about 0.90 and about 1.10 with negligible cementite or other metallic carbide formation.

* * * * *