**TECHNICAL PAPER**

# Independently controlled Carbon and Nitrogen Potential: A new approach to Carbonitriding processes

# Independently controlled Carbon and Nitrogen Potential: A new approach to Carbonitriding processes

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## Abstract

Late research projects show that retained austenite, if stabilized by nitrogen, has a positive influence on the fatigue strength of work pieces. The combined diffusion profile of carbon and nitrogen applied in a carbonitriding process plays the major role, besides the process temperature. Yet today, only the carbon potential is somehow controlled and even this is not easy to achieve.

This paper will present a new system able to measure and control both, the carbon potential and the nitrogen potential independently. The knowledge of the activities of nitrogen and carbon in iron and the effect of alloying elements on such activities as well as the solubilities offers an easy to use method to apply the potentials on real steels.

## Introduction

Gaseous carbonitriding is a termochemical treatment similar to gas carburizing. The difference consists of an addition of ammonia to the process gas which adds nitrogen to the carburized case. The additional nitriding effect is analog to a nitriding process, where the nascent nitrogen is produced by thermal dissociation of the ammonia on the catalytic surface of the work pieces. The nitrogen then diffuses simultaneously with carbon into the steel.

Nitrogen being present in the interstitial profile of carbon has the advantage of an increased hardenability and wear resistance as well as a higher temperature stability of the heat treated parts /1/. As nitrogen stabilizes the retained austenite it also has a positive effect on form stability and fatigue strength of gears /2/. In addition the increased hardenability enables to oil quench machined parts made from unalloyed steels in order to produce strong and wear resistant parts economically.

As the ammonia will eventually completely dissociate into hydrogen and nitrogen at carburizing temperatures and as on the other hand the nitrogen pick-up is driven by the content of still undissociated ammonia, the fundamental problem in a controlled carbonitriding process is that it cannot be performed properly by applying a certain percentage of ammonia in the inlet gas.

The higher the carbonitriding temperature and the higher the combined catalytic surface of load and furnace walls, the faster the ammonia will dissociate. The lower the temperature, the smaller the reactive surface and the higher the total process gas flow at a given ammonia percentage on the inlet, the less ammonia will dissociate and the higher will be the nitriding effect.

The carbon pick-up is as well affected, as with most of the ammonia decomposing, an addition of ammonia to a carburizing atmosphere has the effect of a dilution with nitrogen and hydrogen further affecting the measurement of the carbon potential. In addition the presence of nitrogen in the steel also influences the carbon activity and therefore the carbon pick-up.

## Carburizing and nitriding reactions

Gaseous carbonitriding is performed in an endothermic atmosphere consisting of carbon monoxide, hydrogen and nitrogen and smaller percentages of uncracked hydrocarbons, carbon dioxide and water vapor with an addition of ammonia. Bischoff /3/ presented a graph displaying the concurrently active carburizing and nitriding reactions in this mixture (fig. 1). It can be seen that besides the normal carburizing reactions and the nitriding reaction out of the ammonia dissociation there is also an additional carbonitriding reaction out of cyanide that is created by a reaction of carbon monoxide and ammonia.
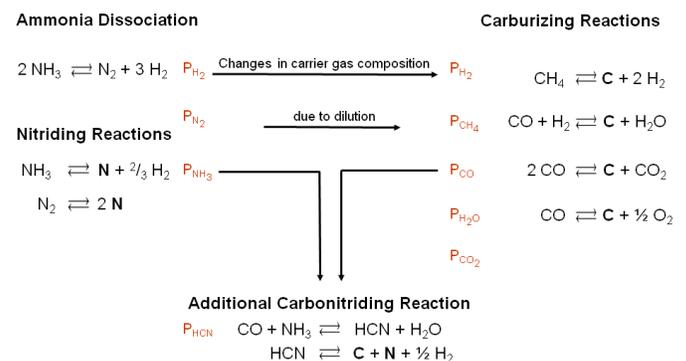


**Ammonia Dissociation**

$2\,NH_3 \rightleftharpoons N_2 + 3\,H_2$   $P_{H_2}$ — Changes in carrier gas composition → $P_{H_2}$

$P_{N_2}$ — due to dilution →

**Nitriding Reactions**

$NH_3 \rightleftharpoons N + {}^2/_3\,H_2$   $P_{NH_3}$

$N_2 \rightleftharpoons 2\,N$

**Carburizing Reactions**

$CH_4 \rightleftharpoons C + 2\,H_2$

$CO + H_2 \rightleftharpoons C + H_2O$   $P_{CH_4}$

$2\,CO \rightleftharpoons C + CO_2$   $P_{CO}$

$CO \rightleftharpoons C + \tfrac{1}{2}\,O_2$   $P_{H_2O}$

$P_{CO_2}$

**Additional Carbonitriding Reaction**

$P_{HCN}$   $CO + NH_3 \rightleftharpoons HCN + H_2O$

$HCN \rightleftharpoons C + N + \tfrac{1}{2}\,H_2$

*Fig. 1: Carburizing and nitriding reactions /3/*

Bischoff [3] as well calculated the varying gas percentages in a carbonitriding atmosphere in equilibrium as a function of temperature (fig. 2).
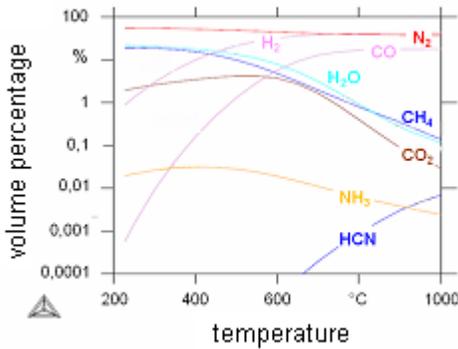


*Fig. 2: equilibrium gas percentages in a carrier gas with 20% CO, 40% $H_2$ and 40% $N_2$ with an $NH_3$ addition of 5% [3]*

It can be seen that with increasing temperature the partial pressure of ammonia decreases whereas the partial pressure of cyanide increases. If we assume the partial pressures being equal at approximately 1650°C (900°C) we would expect a not negligible nitriding effect out of the cyanide reaction.

$$HCN \rightarrow [C] + [N] + H_2$$

A calculation of the nitrogen activities of $NH_3$ and HCN at a temperature of 1650°F (900°C) shows that they would give approximately the same nitriding effect. In reality, as in a carburizing furnace there is a comparably rapid gas exchange the dissociation of the ammonia and therefore as well the calculated equilibrium amount of cyanide will never be reached. Typical fractions of residual ammonia measured in such atmospheres will range between 500 and 5000 ppm, well above the calculated ammonia percentages of less than 100 ppm. Therefore the cyanide reaction will not be taken into account in the further document.

If we further assume that the carburizing will mostly be driven by the heterogeneous water gas reaction

$$CO + H_2 \rightarrow [C] + H_2O$$
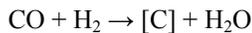
we can simplify the set of carbonitriding formulas to

$$lg(a_{Catm}) = lg(pCO*pH_2/pH_2O) + 7130/T - 7.494 \qquad /4/$$

and

$$lg(a_{Natm}) = lg(pNH_3/p^{1.5}H_2) - 2943/T + 6.196 \qquad /4/$$

All partial pressures are given in bar, temperatures in Kelvin.

Alternatively, if we assume CO, $CO_2$, $H_2$ and $H_2O$ staying in water gas equilibrium, it is possible to use the carbon activity out of the Boudouard reaction

$$2\,CO \rightarrow [C] + CO_2$$
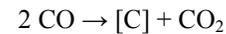
with

$$lg(a_{Catm}) = lg(p^2CO/pCO_2) + 8861/T - 9.107 \qquad /4/$$

Note, that in atmospheres with high residual methane percentages the total carbon activity of the process gas will be affected by the methane reaction and has to be determined by solving the equation

$$J_C = k_{ch4}*(a_{Cch4} - a_{Cfe}) + k_{Boud}*(a_{CBoud} - a_{Cfe}) + k_{het}*(a_{Chet} - a_{Cfe})$$

The factors $k_{ch4}$, $k_{Boud}$ and $k_{het}$ are the carbon transfer coefficients for the three carburizing reactions. These effects have been described in detail by Collin et all [5].

## Interacting activities of carbon and nitrogen

Once carbon and nitrogen is dissolved in the iron lattice they will both influence each others activities.

$$a_{Cfe} = f\,(a_{Catm},\,a_{Nfe})$$

and

$$a_{Nfe} = f\,(a_{Natm},\,a_{Cfe})$$

Using interaction parameters given in table 1 we can calculate the equilibrium carbon and nitrogen weight percentages in a carbonitriding atmosphere.

## (Re-)Definition of carbon and nitrogen potentials

If we define the carbon potential in a carbonitriding atmosphere as the equilibrium surface carbon weight percentage in iron found in addition to nitrogen and if we further define the nitrogen potential as the equilibrium nitrogen weight percentage in iron found in addition to carbon we can use a regular iron shim to determine the total percentage of nitrogen and carbon using a shim weigh. The carbon percentage can then be determined by the combustion method, the nitrogen percentage would be the difference of both measurements.

As nitrogen acts as an alloying element for carbon, the relation between the atmosphere carbon potential and the surface carbon content can be written as:

$$CP = c_{Cequ} = 10^{-0.081 * \%N} * CP_{atm} \qquad /6/$$

and the relation between the atmosphere nitrogen potential and the surface nitrogen content can be written as:

$$NP = c_{Nequ} = 10^{-0.187 * \%C} * NP_{atm} \qquad /6/$$

The atmosphere potentials $CP_{atm}$ and $NP_{atm}$ can be determined by:

$$lg(a_{Catm}) = 2300/T - 2.21 + 0.15*CP_{atm} + lg(CP_{atm}) \;/7/$$

$$lg(NP_{atm}) = lg(pNH_3/p^{1.5}H_2) - 2210/T + 3.91 \ /8/$$
(converted to bar and wt %)

Example:

In a carbonitriding process at 1562°F (850°C) using Endogas with 20 % CO and 40 % $H_2$ with a 5% addition of ammonia and aiming for 0.7 wt% carbon and 0.3 wt% nitrogen in an iron surface the atmosphere potentials come to:

$$CP_{atm} = 0.7/0.95 = 0.74$$

and

$$NP_{atm} = 0.3/0.74 = 0.41$$

This converts to a nitriding potential of

$$K_N = pNH_3/p^{1.5}H_2 = 4.65*10^{-3} \ [bar^{-1/2}]$$

and results into the according partial pressures:

$$pNH_3 = 1.32 * 10^{-3} \ bar = 1323 \ ppm$$

and

$$pH_2 = 0.432 \ bar$$

If we would be aiming for a lower carbon content of 0.6 wt% the $NH_3$ in the exhaust can be reduced to 1267 ppm.

## Solubility and phase transformations

The binary systems for carbon in iron and nitrogen in iron will not reveal the maximum solubility lines of a combined carbon and nitrogen content. The well-known Fe-C or Fe-N phase diagrams typically display carbon and nitrogen in either weight or atomic percentages. Figure 3 combines the two binary systems to a phase diagram using the total nitrogen and carbon contents given in volume percentages, such taking into account that both species will occupy the same interstitial places in the iron lattice.
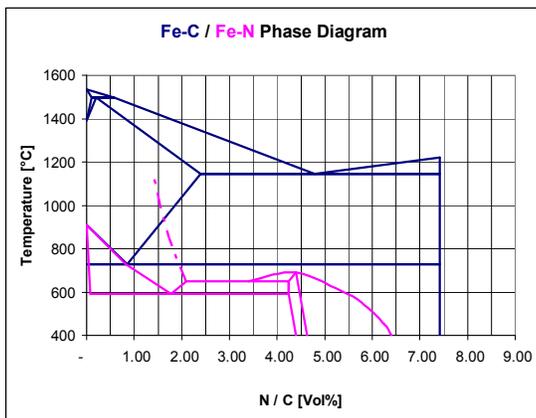


Fig. 3: Combined binary systems Fe-C and Fe-N with C and N given in volume percentages, temperature in centigrade.

It can be derived how the presence of nitrogen expands the existence range of austenite to lower temperatures. The diagram calculates the maximum solubility of nitrogen towards epsilon nitride using the geometric exclusion model given by Slycke and Ericsson /4/. The maximum solubility of carbon towards graphite is taken from standard Fe-C phase diagrams. In absence of nitrogen it can be calculated by setting the carbon activity to 1.

$$lg(a_{Catm}=1) = 2300/T - 2.21 + 0.15* c_{Cgraphite} + lg(c_{Cgraphite})$$

$c_{Cgraphite}$ is also representing the soot limit in the atmosphere.

As this boundary will be affected by the presence of nitrogen it might approximately be adjusted by:

$$c_{Cmax} = c_{Cgraphite} * 10^{-0.103* \%N}$$

The maximum solubility of nitrogen towards epsilon between 1562°F (850°C) and 1652°F (900°C) can be calculated as:

$$c_{Neps} = 10^{464.28/T - 0.050394} - 0.02$$

and further adjusted to the presence of carbon by:

$$c_{Nmax} = c_{Neps} * 10^{-0.14* \%C}$$

## Influence of alloying elements

Both, the activity of carbon and nitrogen will also be affected by the alloying elements being present in regular steel. For control purposes this effect will be taken into account by applying alloying factors for both, carbon and nitrogen. The alloying factors are following the definition:

$$kj = c_{Steel} / c_{Fe}$$

with

$$lg(c_{Fe}/c_{Steel}) = sum \ of \ (c_i * e_{ji})$$

for j = {C, N} and i = {C, N, ... }, $c_i$ in wt%. The according values for $e_{ji}$ can be taken from table 1.

The equilibrium carbon content in alloyed steel exposed to an atmosphere carbon potential will come to:

$$c_{C-Steel} = k_C * CP_{atm}$$

with

$$lg(k_C) = - (0.010*B\% + 0.081*N\% - 0.014*Al\% \ ...)$$

The equilibrium nitrogen content in alloyed steel exposed to an atmosphere nitrogen potential will come to:

$$c_{N-Steel} = k_C * NP_{atm}$$

with

$$lg(k_N) = -(0.187*C\% + 0.124*Si\% \ ...)$$

*Table 1: Interaction parameters of alloying elements on the activity coefficients for Carbon and Nitrogen in gamma iron at 1652°F (900°C), average values taken from literature /6/.*

| J | $e_{Ci}$ | $e_{Ni}$ |
|---|---|---|
| B | 0.070 | (unknown) |
| C | - | 0.187 |
| N | 0.081 | - |
| Al | -0.014 | (unknown) |
| Si | 0.055 | 0.124 |
| P | 0.130 | 0.050 |
| S | 0.140 | 0.017 |
| Ti | -0.194 | (unknown) |
| V | -0.210 | -0.523 |
| Cr | -0.043 | -0.158 |
| Mn | -0.016 | -0.041 |
| Co | 0.012 | 0.020 |
| Ni | 0.014 | 0.022 |
| Cu | -0.006 | (unknown) |
| Nb | -0.137 | -0.358 |
| Mo | -0.015 | -0.080 |
| W | -0.023 | -0.037 |

The boundaries towards graphite and cementite formation can be estimated by:

$$c_{Cmax\text{-}Steel} = k_g * c_{Cgraphite} - MC/MB * c_{Boron}$$

with

$$lg(k_g) = sum (c_i * e_{Ci}) - c_N * 0.103$$

and i not in {B, N, P, S}, as these elements are interstitials like carbon. Boron is able to replace up to 80% of carbon in cementite, therefore the boron is treated like carbon, taking the different weights into account (MC = 12.011, MB = 10.81).

The boundary towards $Fe_{2\text{-}3}[N+C]$ epsilon can be estimated by:

$$c_{Nmax\text{-}Steel} = k_e * c_{Neps}$$

with

$$lg(k_e) = sum (c_i * e_{Ni}) - c_C * 0.14$$

and i not in {C, B, P, S}.

Exceeding this boundary will force the formation of meta stable nitride, causing pores in the parts surface layer /9/ by the possible back reaction:

$$Fe_{2\text{-}3}N \rightarrow Fe + N_2$$

## Measuring the atmosphere potentials

According to DIN 17 022 Part 3 /7/ the carbon potential of a carburizing atmosphere can be calculated out of the mV signal of an **OXYGEN PROBE** (following the heterogeneous water gas reaction) by:

$$E = 0.0992*T*(lg\ pCO - 1.995 - 0.15\ CP - lg\ CP) - 816.1\ [mV]$$

As the partial pressure of carbon monoxide is typically used as a constant number given by the carrier gas, in a carbonitriding atmosphere either the dilution caused by the ammonia addition has to be taken into account or the actual partial pressure of CO has to be measured using an analyzing instrument. Under regular conditions at atmospheric pressure, where all of the ammonia will be nearly dissociated it is sufficient to use:

$$pCO_{atm} = vol\%\ CO_{carrier} / (100 + 2*NH_{3\text{-}add})$$

One method to determine the nitrogen potential is to measure the partial pressure of ammonia using an analyzer calibrated to a range of 0 – 10000 ppm. The hydrogen partial pressure may then be measured with a second analyzer or estimated as

$$pH_{2\text{-}atm} = (vol\%\ H_{2\text{-}carrier} + 1.5*NH_{3\text{-}add}) / (100 + 2*NH_{3\text{-}add})$$

A second possibility is the direct measurement of carbon and nitrogen in equilibrium in iron using a wire-sensor /10/. This type of sensor exposes a thin iron wire (see figure 4) to the carbonitriding atmosphere. As both, carbon and nitrogen will be interstitially dissolved in the iron lattice, the electrical resistance of the wire changes (see figure 5).
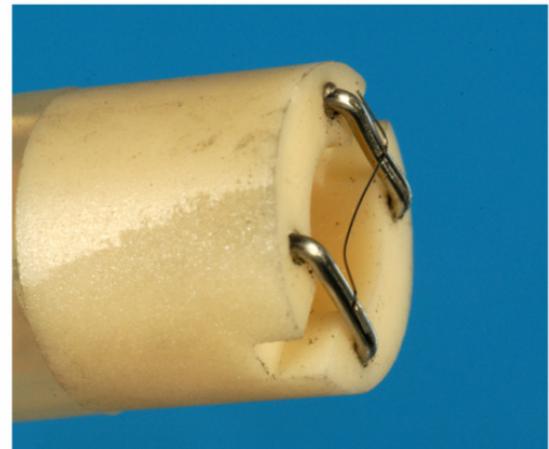


*Fig. 4: Active element of the wire sensor*

Chatterjee-Fischer /11/ is giving the relation for nitrogen induced increase of resistivity versus carbon induced as:

$$\Delta R_C = 0.7 * \Delta R_N$$

In order to achieve the 0.7 wt% C plus 0.3 wt% N in the example above, the wire would have to be controlled to a

[N+C] content of 0.94 wt% virtual carbon. The carbon potential controlled with the oxygen probe has to be adjusted to 0.74 %C and the atmospheric nitrogen potential would be at 0.41 %N.
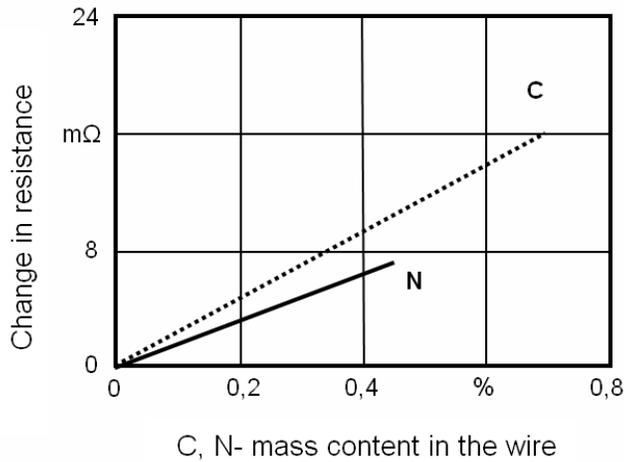


*Fig. 5: Induced resistance change in an iron wire due to carbon and nitrogen uptake /11/*

Figure 6 shows the computed signals of an oxygen probe and a wire sensor exposed to a carburizing atmosphere. The moment ammonia is added to the process gas the wire's combined carbon and nitrogen potential increases while the carbon potential measured with the oxygen probe and adjusted for the dilution stays level.
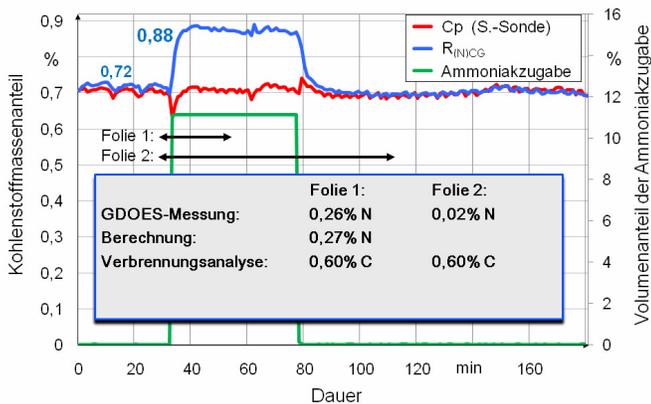


*Fig. 6: Nitriding and de-nitriding of wire sensor and iron shims at 1562°F (850°C). The red line indicates the controlled carbon potential (oxygen probe); the blue line displays the increase in virtual carbon [N+C] in the wire. The green line shows the ammonia percentage in the gas inlet /3/*

Iron shims (Folie 1 and 2) verified the nitrogen content, the carbon content was lower as expected.

## Carbon and nitrogen uptake

Based on the assumption of the heterogeneous water gas reaction being the dominant carburizing reaction, the carbon uptake can be determined by the partial pressures of carbon monoxide and hydrogen in the process atmosphere shown in figure 7. The curve can be approximated by:

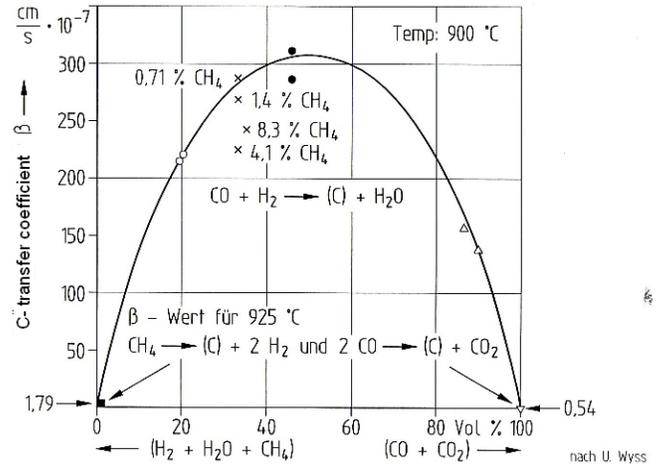$$\beta_C = 0.00018*pCO*pH_2 - (pCO*pH_2/65.78)^2 \text{ [cm/s]}$$



*Fig. 7: Carbon transfer coefficient beta as a function of the volume percentages of carbon monoxide and hydrogen at atmospheric pressure and 1652°F (900°C) /12/*

While CO will be diluted whereas $H_2$ will increase by the dissociated ammonia, the transfer coefficient will be affected very little. An ammonia addition of 10 % decreases the beta coefficient only by 4.5 %.

In a carbonitriding process the nitrogen activity given by the ammonia dissociation is greater than the activity given by the partial pressure of nitrogen. Consequently the equilibrium nitrogen content for $pNH_3/p^{1.5}H_2$ is as well much higher. Therefore there has to be a local equilibrium of a dissociation and a formation of ammonia on the parts surface. As the nitrogen uptake is limited by the denitriding reaction it can be determined by the partial pressure of hydrogen:

$$\beta_N = 0.09*e^{-64247/RT}*pH_2 \text{ [cm/s]} \qquad /13/$$

The carbon and nitrogen flux into the surface can be expressed as:

$$J_C = \beta_C * (c_{Cequ} - c_{C\text{-surface}}) \text{ [wt\%*cm/s]}$$

and

$$J_N = \beta_N * (c_{Nequ} - c_{N\text{-surface}}) \text{ [wt\%*cm/s]}$$

## Carbon and nitrogen diffusion

The carbon diffusivity in γ-iron is a function of temperature and dissolved carbon.

$$D_C = e^{-(18900/T+0.38)} * e^{(4300/T-2.63)*(\%C^{1.5})} \text{ [cm}^2\text{/s]} \qquad /14/$$

As both agents are occupying the same interstitial places one can assume that the diffusivity of carbon and nitrogen will be dependent on the actual amount of nitrogen and carbon /4/ already dissolved and causing the lattice to open. As a simplification, nitrogen will be treated as "virtual carbon", applying a factor taking the different dimensions into account.

$$D_C = e^{-(18900/T+0.38)} * e^{(4300/T-2.63)*((\%C+0.62*\%N)^{1.5})} \, [cm^2/s]$$

Based on a relation given by Slycke /4/ the nitrogen diffusion coefficient of nitrogen between 1562°F (850°C) and 1652°F (950°C) can be adjusted to:

$$D_N = 0.45*e^{-(18900/T+0.38)} * e^{0.62* (4300/T-2.63)*((\%C+0.62*\%N)^{1.5})} \, [cm^2/s]$$

Figure 8 displays the result of a simulation with HT-Tools using the above equations. Carbon and Nitrogen profiles are calculated. Based on the carbon and nitrogen content the software calculates the expected hardness curve after quenching and regular tempering.
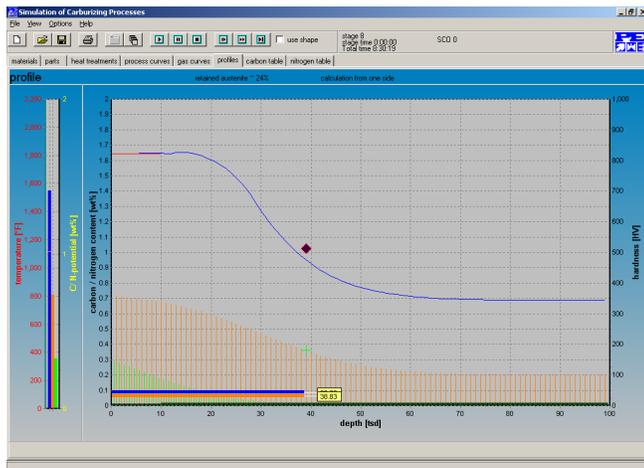


*Fig. 8: C- and N- profiles in a steel bar made from AISI 8620 aiming for a case depth of 40 thousands and a surface carbon content of 0.72 %C and a surface nitrogen content of 0.30 %N*

## Controlling the potentials

In our tests we have been using a PROTHERM 500 programmable controller, able to control temperature and atmosphere.

Like a normal carbon potential controller, the system uses propane or methane as enrichment gas and air for reducing the carbon potential. The nitrogen potential is controlled by increasing or decreasing the ammonia flow.

The controller is able to either calculate the dilution, if the flows are known, or it can use the signals of connected analyzers to adjust carbon monoxide and hydrogen. For determining the nitrogen potential, the controller can be connected to a wire sensor or to an ammonia analyzer.

The recipe holds the composition of the steel to be treated calculates the alloying factors for carbon and nitrogen and

calculates the carbon and nitrogen diffusion during the process based on the actual carbon and nitrogen transfer coefficients and the diffusion coefficients at temperature and carbon and nitrogen content throughout the calculated profiles.

Temperature control offers RAMP HEAT, HEAT, HOLD, RAMP COOL, the atmosphere control offers RAMP CP/NP, HOLD CP/NP, and several special functions, SOOT LIMIT, AUTO BOOST, $Fe_{2-3}N$ LIMIT and SURFACE CONTENT N/C.

In order to achieve short process times, the proposed atmosphere control starts with a boost stage maintaining high potentials. During the AUTO BOOST the controller limits the atmosphere carbon potential to a set percentage of the soot limit (see above).

$$CP_{atm} <= AUTO \ FACTOR * c_{Cmax}$$

The moment the calculated surface carbon content in alloyed steels reaches the maximum solubility towards graphite, adjusted to the actual surface nitrogen content, the atmosphere carbon potential will automatically be reduced (see figure 9).
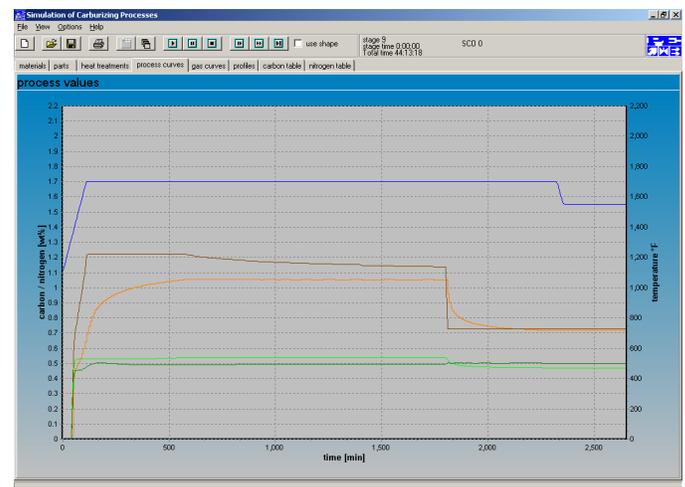


*Fig. 9: Chart recorder displaying the controlled atmosphere potentials for carbon (brown) and nitrogen (light green), the temperature (blue) and the calculated surface contents for carbon (orange) and nitrogen (dark green).*

The desired nitrogen content in the surface was given as a setpoint of 0.5 wt% N in the steel. The ammonia addition is automatically adjusted to achieve the necessary $NP_{atm}$.

The end condition for an auto boost stage can be either time or a set percentage of the desired carburizing depth.

In the diffusion stage the controller will set the atmosphere potentials automatically according the DESIRED SURFACE CONTENTS of CARBON and NITROGEN. This stage will typically end once the desired carburizing depth is reached.

It is also possible to use the atmosphere potentials $CP_{atm}$ and $NP_{atm}$ as setpoints, in which case no automatic limitations to soot, cementite or epsilon will be performed. This represents a more classical process variant.

## Test Results

Tests done at the IWT and several customers show a good correlation between predicted and measured carbon and nitrogen contents in samples (see figures 10 and 11).
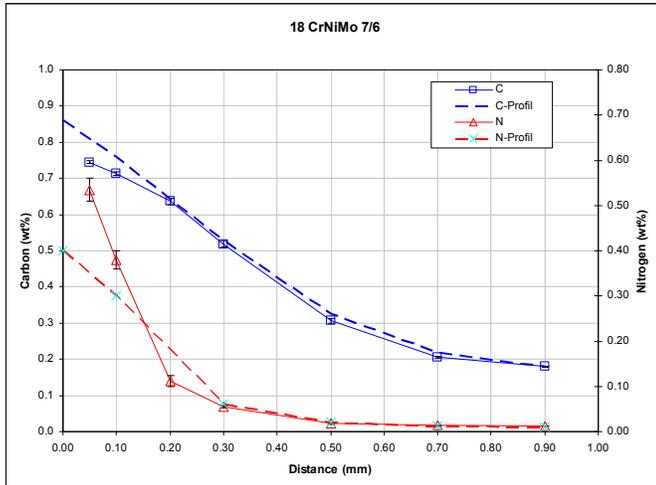


*Fig. 10: Measured (C, N) and calculated (C-Profile, N-Profile) carbon and nitrogen contents in an 18 CrNiMo 7/6 test piece, carbonitrided at1544°F ( 840°C)  for 280 minutes.*
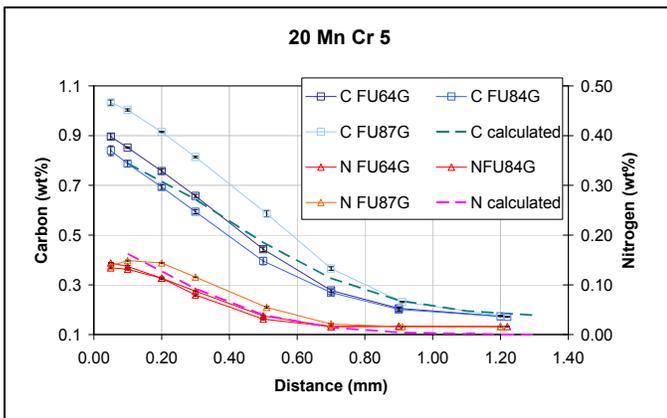


*Fig. 11: Measured and calculated carbon and nitrogen contents in 20 MnCr 5 test pieces, carbonitrided at 1706°F (930°C)  for 240 minutes.*

## Conclusions

Incorporating the results of late research projects into a process controller enables a new attempt to performing carbonitriding processes.

The ability to measure and control both, the carbon and nitrogen potential in the atmosphere takes the uncertainty of the ammonia dissociation, caused by changing surface conditions out of the equation. Besides obvious advantages in the process control and the ability to reproduce batch after batch, this will also be valid if furnaces are used for carburizing inped carbonitriding processes where the NP control can shorten formation times.

Using the interaction parameters to adjust the atmosphere potentials to the varying steel compositions enables a controlled carbonitriding treatment to desired surface contents of carbon and nitrogen which might open a new field of investigations on working properties of parts.

The inbuilt safety functions to avoid cementite and epsilon formation enables the use of higher potentials during boost stages, aiming for shorter process times while at the same time being safe not to create a porous layer.

The online diffusion calculation for carbon and nitrogen enables a treatment to the specification such as case depth and surface carbon and nitrogen content.

## Acknowledgments

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