

TECHNICAL PAPER

State-of-the-Art
Controlled Nitriding & Nitrocarburizing

State-of-the-Art Controlled Nitriding and Nitrocarburizing

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Abstract

The process of gaseous nitriding is considered to be fully controllable by temperature and the nitriding potential. Most of the commercially used control systems are based on the Lehrer Diagram showing the relation between nitrogen-iron phases, temperature and the partial pressure ratio of ammonia and hydrogen. This is also reflected in the measuring equipment used to determine the nitriding potential.

As everyone knows, Lehrer's phase diagram was created out of a reaction between a set gas mixture of ammonia and hydrogen and pure iron. In industrial nitriding cycles, dealing with real materials and real parts the results often do not match the expectations.

The presentation will try to explain the parameters that have to be taken into account and how to measure, set and control the according potentials by giving an insight on the available equipment.

Keywords

Atmosphere potentials, measuring equipment, control policies, relation between potentials and nitrided layers

1 Adjusting the Lehrer Diagram to Steels

Controlling nitriding processes is mostly done by controlling the nitriding potential $K_N = p_{NH_3}/p_{H_2}^{3/2}$ of the furnace atmosphere in order to achieve the desired results in terms of diffusion depth as well as thickness and composition of the surface layer. Technically spoken we like to reach a certain nitrided hardness depth and a white layer of either Fe_4N Gamma Prime or $Fe_{2-3}N$ Epsilon with a minimum and maximum thickness. The relation between temperature, nitriding potential and the iron-nitrogen phase is given by the Lehrer Diagram /1/ (see figure 1). In addition often the porosity of the white layer should not exceed a certain percentage.

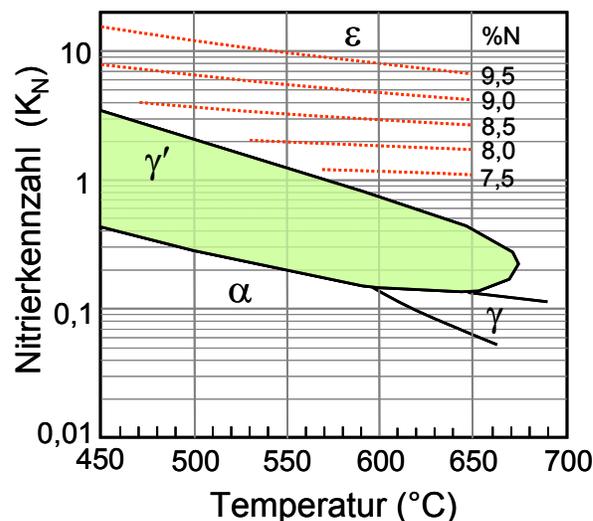


Figure 1: p_{N_2} , temperature, Fe-N Lehrer Diagram /1, 2/

In industrial performed nitrocarburizing processes still either no potential at all or, if any, the nitriding potential is the preferred potential to be measured and controlled. But adding carbon bearing gases such as carbon dioxide, carbon monoxide, endogas and in some cases hydrocarbons has a huge impact not only on the result but also on the Lehrer Diagram. This impact is given by the additional carbon activity of the process gas which can be determined by measuring either the carburizing potential $K_{CB} = p^{CO}/p^{CO_2}$ out of the Boudouard reaction or the carburizing potential $K_{CW} = p^{H_2} \cdot p^{CO}/p^{H_2O}$ out of the heterogeneous water gas reaction. Naumann and Langenscheid /3/ investigated into the influence of the existence of both, nitrogen and carbon on the iron-nitrogen-carbon phases and plotted a set of diagrams for different temperatures (see Figure 2).

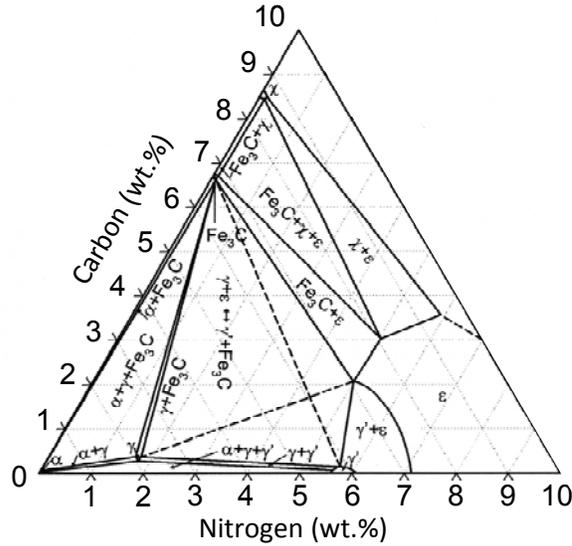
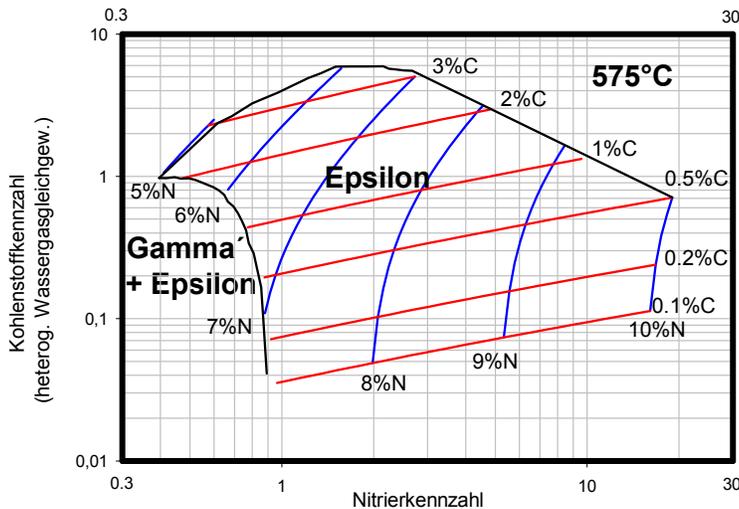


Figure 2: wt%N, wt%C, Fe-N-C Phase Diagram at 575°C /3/



(NICEPS5.JNB)
(NC4.XFM)

(Phasengrenzen gem. Naumann / Langenscheid, Isokonzentrationsl. gem. Kunze)

Figure 3: Nicarm Diagram /4/

Unfortunately these diagrams cannot be used for control purposes and have to be converted into Fe-N-C phase diagrams depending on temperature and potentials. Based on a set of formulas published by Kunze /4/, Weissohn /5/ developed a specialized Nitrocarburizing Diagram (see figure 3).

This diagram shows the phase composition on the top of the white layer we would create if we treated pure iron in an atmosphere with set K_N and K_{CW} at a given temperature. Varying the temperature throughout the process obviously would require a set of diagrams to adjust for the influence of the temperature. In addition we have to face the fact that most systems are not equipped in a way to enable a control of two potentials at the same time anyways.

There is another aspect we have to take into account, which is actually the most important aspect of all: we never treat pure iron! By solving those equations backwards and applying the known activity coefficients we can plot a modified Lehrer Diagram (Figure 4) adjusting for the carbon content of the steel to be treated. The phase boundaries are calculated using equations given by Maldzinski /6/.

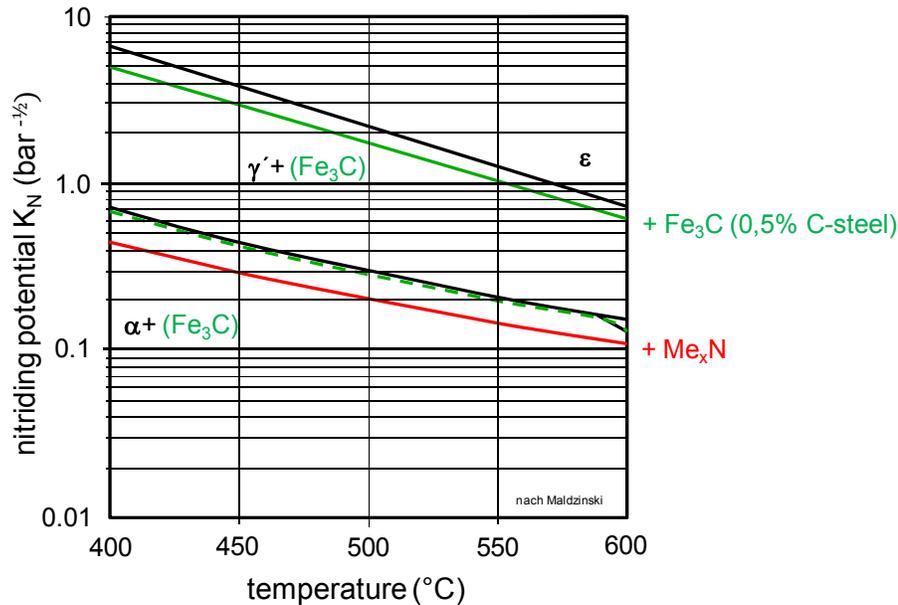


Figure 4: Lehrer Diagram adjusted to Fe + 0.5 wt%C adjusted to AISI 4140 steel

Figure 4 shows that the presence of 0.5 wt% carbon in iron does not have a huge impact on the alpha to gamma prime boundary but the presence of carbon enables to build epsilon $Fe_{2-3}NC$ already at a deliberately lower nitriding potential. More generally spoken, nitriding high carbon steel is very similar to nitrocarburizing low carbon steel.

In carburizing the effects of alloying elements are well known and in regular use in the daily work by applying the alloying factor to predict the carbon percentage in the surface but also to predict the shifting of the S'-E' and S-E lines giving the upper limit of carbon solubility towards graphite and Fe_3C cementite.

Figure 4 also shows the Lehrer Diagram modified to fit a regular AISI 4140 steel. The presence of the other alloying elements such as silicon, manganese, chromium and molybdenum is reflected in an additional alpha to Me_xN_y line.

2 Taking the Nitrogen Transfer Speed into Account

When Jung /7/ developed a controlled high pressure nitriding process he was using the effect that with increasing furnace pressure but keeping the nitriding potential constant the partial pressure of ammonia and by the power of 1.5 the partial pressure of hydrogen increases as well. By applying an equation given by Grabke /8/ explaining the nitrogen transfer into alpha iron he was able to calculate the relative nitrogen transfer speed depending on temperature and pressure. Jung was able to show that the thickness of the white layer increases with higher pressures and therefore higher partial pressures of hydrogen.

Total Pressure	Hydrogen Partial Pressure	Relative Nitrogen Transfer Speed
1 bar	0.33 bar	0.30
4 bar	0.94 bar	1.00
8 bar	1.57 bar	1.67
12 bar	2.11 bar	2.24

Table 1: hydrogen partial pressures and relative reaction speed at 570°C, $K_N=3 \text{ bar}^{-1/2}$ for varying total pressures (Jung /7/)

Table 1 might not be too interesting for regular atmospheric nitriding but the dependence on pressure also works into the direction of lower pressures or so-to-speak diluted atmospheres. Especially in big furnaces the use of nitrogen diluted ammonia in order to enable lower potentials is a common practice. However, diluting down too much causes unwanted effects like non-uniform layers. Calculating a second table (table 2) as an expansion to Jung into lower total pressures will give us an idea of what to expect in terms of the nitrogen transfer speed and white layer growth.

Total Pressure / Nitrogen- Dilution	Hydrogen Partial Pressure	Relative Nitrogen Transfer Speed
1 bar / 0 %	0.33 bar	0.30
0.8 bar / 20 %	0.28 bar	0.23
0.5 bar / 50 %	0.19 bar	0.13
0.2 bar / 80 %	0.09 bar	0.04

Table 2: hydrogen partial pressures and relative reaction speed at 570°C, $K_N=3 \text{ bar}^{-1/2}$ for nitrogen diluted atmospheres

The effect of dilution has been studied by Zimdars /9/ and his tests showed that a dilution of an oxi-nitriding atmosphere ($K_N=3$, $K_O=p\text{H}_2\text{O}/p\text{H}_2=0.27$) down to 15% NH_3 in the exhaust did not slow down the layer growth dramatically. Diluting further resulted in much thinner white layers as well as more shallow cases. 15% of residual ammonia can be expressed as a 50% dilution with nitrogen or a vessel pressure slightly below 0.5 bar.

Please note that Zimdars did a nitriding process with oxygen addition. Chatterjee-Fischer /10/ reported that an oxygen addition to a nitriding atmosphere accelerates the growth of the white layer. This effect was explained by the reduction of the hydrogen partial pressure due to the formation of water vapour and therefore the increasing nitriding potential. Zimdars on the other hand controlled both potentials excluding the thought effect. Therefore the influence of oxygen can be better explained by its ability to remove the hydrogen atoms sticking to the steel surface and hindering the next ammonia molecule to be attached and dissociated. The same effect can be seen in nitrocarburizing atmospheres containing CO and CO_2 .

Both, Jung and Zimdars also report a strong dependency of the layer growth on the steel composition.

As a consequence, starting a nitriding process with a nucleation stage building a tiny white layer feeding the further nitrogen diffusion avoids non-uniform layers aiming for deep cases and a restricted or no white layer at all.

3 Relation between Activities, Potentials and Partial Pressures

In order to control the process we first have to identify what gases we have to measured in the furnace atmosphere. In general the influencing parameters besides the temperature are the nitrogen activity a_N and the carbon activity a_C . As the nitrogen activity in a nitriding process is proportional to the nitriding potential by the relation

- $a_N = K_1 \cdot K_N$ with $K_N = p\text{NH}_3/p\text{H}_2^{3/2}$

we need to be able to determine the partial pressures of ammonia and hydrogen. In the control system a conversion between nitrogen activity and nitriding potential is not necessary as the nitrogen activity in the steel surface can also be expressed as an "inner" nitriding potential.

The influence of carbon on the process cannot be expressed in such an easy manner. Depending on the carbon bearing gas used and depending on the measuring system on hand it is more practical to use the activity instead of the varying carburizing potentials. The relation between the carbon activity and the carburizing potentials are as follows:

- $a_C = K_2 * K_{CB}$ with $K_{CB} = p^2CO/pCO_2$
- $a_C = K_3 * K_{CW}$ with $K_{CW} = pH_2 * pCO/pH_2O$
- $a_C = K_4 * K_{C-O_2}$ with $K_{C-O_2} = pCO/pO_2^{1/2}$
- $a_C = K_5 * K_{C-CH_4}$ with $K_{C-CH_4} = pCH_4/p^2H_2$

Therefore the measured potential has to be converted to the controlled potential or the carbon activity is the controlled parameter.

Simply put we have to be able to measure

- ammonia and hydrogen in nitriding processes
- plus water vapour or oxygen in oxinitriding processes
- plus carbon monoxide and either carbon dioxide, water vapour or oxygen in nitrocarburizing processes.

The necessity to determine the carbon activity from hydrocarbons is very seldom due to the fact that these molecules will crack very slowly at the usual process temperatures ranging from 500°C to 590°C. There are some special processes applying i.e. propane, other processes operate at higher temperatures, but in general carbon oxides are the most used additions.

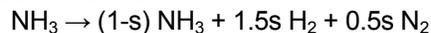
K_1 through K_5 are thermodynamical constants depending on temperature /10/.

4 Commonly used Measuring Equipment

Most industrial installations capable of potential control use devices to measure hydrogen or oxygen or a combination of both. If the gas flows are known measuring the hydrogen partial pressure respectively the hydrogen percentage in the exhaust at a known pressure is giving the nitriding potential out of the ammonia dissociation.



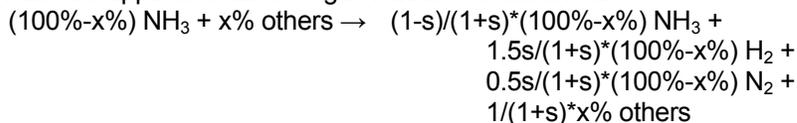
As we try to maintain a certain nitriding potential only a part of the inlet ammonia is dissociated. Assuming a dissociation rate of s ($0 \leq s \leq 1$) the equation comes to



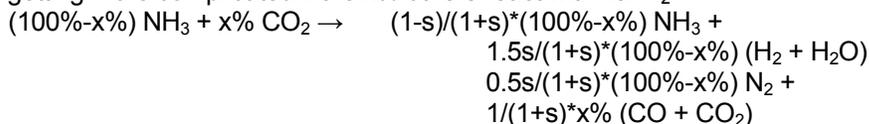
Or giving the percentages



This can also be applied to different gas mixtures in which case



and it is getting more complicated if the $x\%$ others react with i.e. H_2

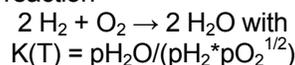


The ratio between H_2 and H_2O as well as the ratio between CO and CO_2 follows the water shift reaction when in equilibrium

$$K_W = (pCO_2 * pH_2) / (pCO * pH_2O)$$

with K_W being a thermodynamical constant depending on temperature.

By using a hydrogen analyzer to determine the H_2 percentage and an oxygen probe to determine the ratio of pH_2O/pH_2 according to the equilibrium of the reaction



in combination with the Nernst equation to determine the oxygen partial pressure

$$EMF = 0.0496 * T * \lg(pO_2/0.209) [mV] /10/$$

it is possible to calculate the dissociation degree s and further on K_N .

There are other attempts to determine K_N . One uses the effect of ammonia dissociating to hydrogen and nitrogen by doubling its volume. This method requires a furnace that can be closed on the gas inlet and the exhaust by valves. By cyclically sealing off the furnace and measuring the pressure over a specified time the pressure increase corresponds to the dissociation degree s with a known ammonia content /12/.

Using two hydrogen analyzers with a dissociator in between enables the direct measurement of ammonia and hydrogen and therefore K_N with a small error that will be corrected by the use of an additional oxygen sensor /13/. Obviously it is also possible to use an ammonia analyzer i.e. an infrared (IR) analyzer to determine the ammonia percentage and either derive the hydrogen percentage in a similar manner as when using a hydrogen analyzer or simply by using an additional hydrogen analyzer. The disadvantage in using an IR ammonia analyzer is that the sampling gas has to be dried before entering the measuring cell. The NH_3 absorption lines are in the middle of water absorption lines with the water giving a much higher signal compared to the ammonia. Drying the gas, in other words filtering the water vapour percentage causes pretty high errors in the measured K_N in a nitrocarburizing or oxinitriding atmosphere.

For the determination of the carbon activity it is either possible to use the equilibrium constant K_W if the total flow of CO and CO_2 is known, derive the ratio from an oxygen probe signal or to measure one or both gas percentages in the exhaust using an infrared analyzer. This would give the a_C out of the Boudouard reaction, but we have to take into account that the water gas reaction is much faster. Therefore in order to determine the most probable conditions a measurement of CO and using the already measured H_2 to H_2O ratio is providing a more accurate result.

In many cases a nitrocarburizing process is followed by a post-oxidation stage in order to improve the corrosion resistance. The iron-oxygen phase aimed for is magnetite. The phase can be controlled by the oxidation potential $K_O = p_{H_2O}/p_{H_2}$ which can be directly derived from an oxygen probe.

5 Controlling the Potentials

Controlling the nitriding potential in a nitriding process is fairly easy. Depending on the thermal and catalytic dissociation given by process temperature and reactive surface (figure 5) the residual ammonia content has to be kept at a level maintaining the desired setpoint of K_N .

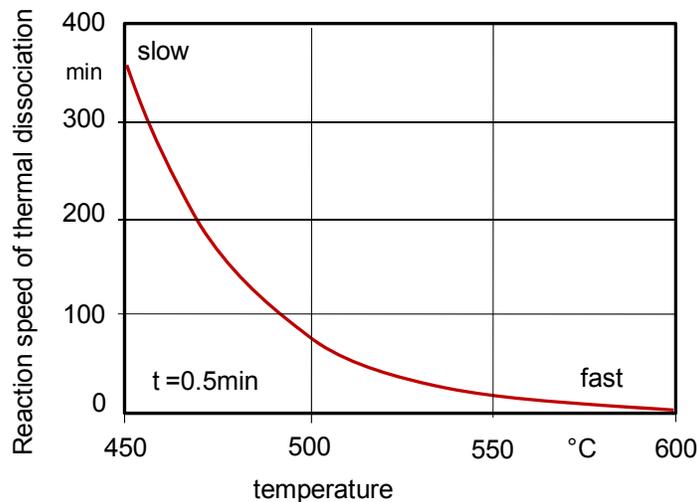


Figure 5: Reaction speed of the thermal dissociation of ammonia as a function of temperature for a given reactive surface

Where at high temperatures and big surfaces the dissociated amount of ammonia has to be replaced by fresh ammonia in the inlet, at lower temperatures and small surfaces either the total flow of ammonia can be reduced or a part of the ammonia has to be replaced by pre-dissociated ammonia or both. Especially in big vessels where big

parts (= small surface) are treated to deep cases often the ammonia will be diluted with nitrogen to reduce costs and pollution. This can be done only to a certain limit as shown above in order not to risk non-uniform diffusion layers. Nitrocarburizing processes are still mostly performed by adding either CO₂ or endogas with a set percentage of the total flow. This will not enable a true control of the white layer composition. In order to control both parameters, K_N and a_C the flow and mixture of the carbon oxides have to be adjusted accordingly.

Figures 6 /14/ show the K_N-K_{CB} (converted from a_C) control range for a variety of fresh gas mixtures.

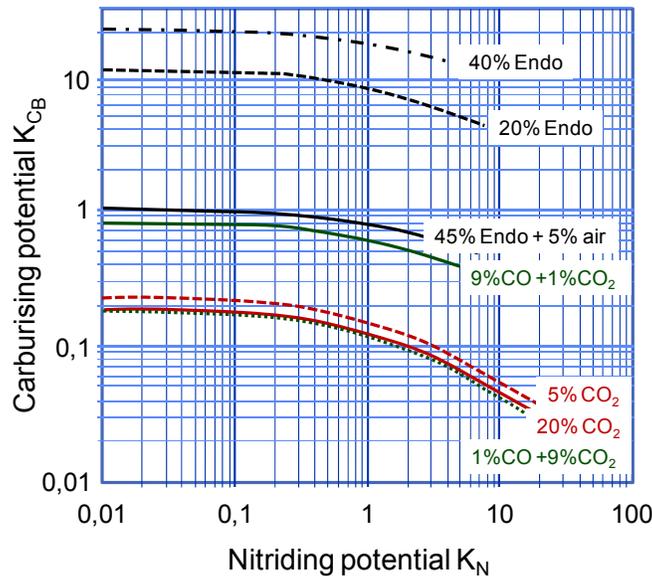


Figure 6: K_N-K_{CB} control range at 580°C using NH₃/CO₂ mixtures using NH₃/Endo (20%CO)/Air mixtures using NH₃/CO/CO₂ mixtures

The easiest way to control the carbon activity is given by using CO and CO₂ and adjusting the ratio at a constant total percentage of the inlet gas. First it gives a wide range in a_C and second, even more important, it does not influence the K_N control loop too much. Using endogas for a_C control will always lower the maximum possible nitriding potential due to its high hydrogen content. Carbon dioxide by itself is not useful for an a_C control. As the CO₂ first has to be reduced to CO before building a carbon activity ($a_C = K \cdot p^2 \text{CO} / p \text{CO}_2$) the control behaviour is reversed. Actually adding more CO₂ results in lower a_C and vice versa. Therefore when trying to get a maximum a_C the CO₂ on the inlet would have to be nearly closed in which case the total amount of carbon in the furnace atmosphere would have a high potential but on the other hand there would be nearly no carbon at all – a high activity but no availability.

For the control of the post-oxidation stage in most cases an injection of either water or in some cases NO₂ is used to build-up the potential. For control purposes it is an advantage to add a small amount of hydrogen to have a counter weight to the oxygen. This can be done in a safe way by using ammonia, dissociated ammonia or pure hydrogen.

6 Control Variants

Depending on the specification to be obeyed still the most popular variants are:

- Fixed flows, no potential control
- Dissociation control (DIN 17 022-4, SAE AMS 2759/6A)
- K_N control (DIN 17 022-4, SAE AMS 2759/10)

Please note that the dissociation degree in the DIN specification $DG = (100\% - \text{NH}_3 \text{ in exhaust}) / (100\% + \text{NH}_3 \text{ in exhaust})$ does not match the AMS specification defining the dissociation by $D = 100\% - \text{NH}_3 \text{ in exhaust}$ read by a burette.

For nitrocarburizing aerospace parts there is a relatively new specification setting the range of white layer porosity that requires

- K_N-K_{CB} control (SAE AMS 2759/12)

In order to reduce process time more and more nitriding processes are converted to oxi-nitriding processes requiring

- K_N - K_O control

But there are also some more modern control variants aiming for white layer properties such as

- Phase control
- Weight percentage control

Phase control is aiming for a specific iron-nitrogen phase set by a percentage between alpha Fe – Fe₄N – Fe₂₋₃N – Fe₂N automatically adjusting the K_N throughout the process. This results in very uniform layers with restricted porosity /18/.

The setpoints for an atmosphere controller operating in weight percentage control mode are given in wt% N and wt% C in the white layer, the according process parameters K_N and K_C respectively a_C are adjusted automatically throughout the stage.

7 Controlled Nitrocarburizing Experiments

Based on measuring the partial pressures of hydrogen and oxygen a control cycle for nitriding and carburizing potential was built up on a nitriding furnace /14/. Figures 7 and 8 show the controlled and measured variables in a nitrocarburizing process at a temperature of 570 °C and with a duration of 4 h. The potentials for this example were chosen as nitriding potential $K_N=1$ and carburizing potential $K_{CB}=0.3$. For K_N control a hydrogen probe was used. The residual ammonia content was additionally checked by an infrared analyzer in the exhaust. To keep the residual ammonia content according to set K_N a mixture of fresh and pre-dissociated ammonia was used. Figure 7 shows the plotted gas flows of fresh and pre-dissociated ammonia. The nitriding potential could be kept at the setpoint of 1. Controlling K_{CB} was done by additionally measuring the oxygen partial pressure with an oxygen probe. As mentioned above, using mixtures of CO and CO₂ does influence the K_N control loop less than using other carbon-containing gas mixtures thus this way of controlling carbon activity was chosen. Figure 8 shows the plotted gas flows of CO and CO₂. The K_{CB} level of 0.3 was kept constant during the nitrocarburizing treatment.

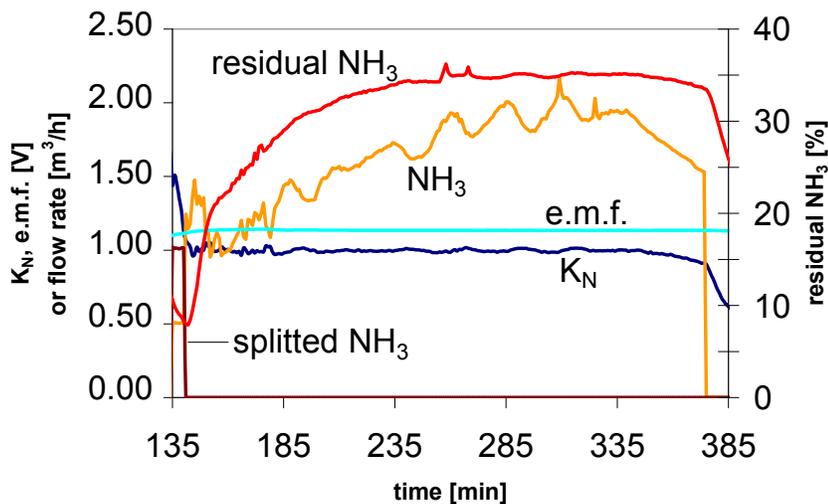


Figure 7: K_N control for nitrocarburizing process 570 °C, 4 h, $K_N=1$, $K_{CB}=0.3$

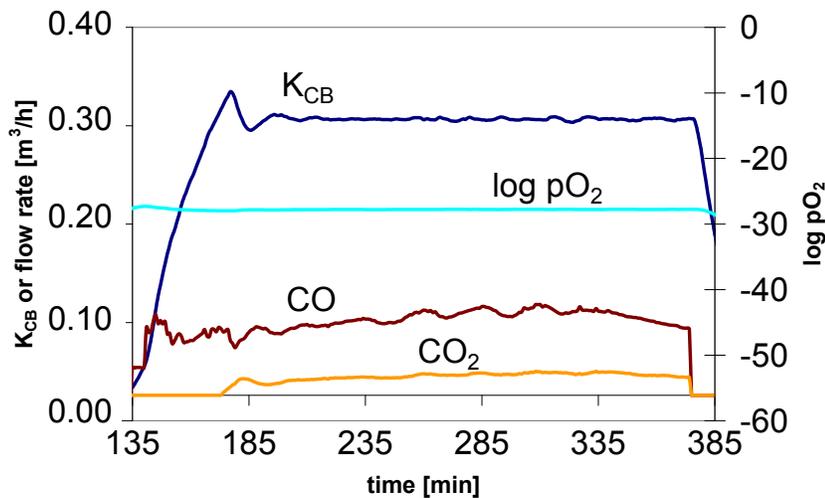


Figure 8: (K_{CB} control for nitrocarburizing process 570 °C, 4 h, $K_N=1$, $K_{CB}=0.3$)

In the above discussed nitrocarburizing process the steels AISI 1015, 1045, 1060, 4140 and H13 were treated. The results (see table 3) were compared to calculated phase diagram in figure 3. Note that the alloying elements influence the lines in the Fe-N-C phase diagram. Thus the N and C contents obtained from GDOS depth profile at a depth of 0.5 μ m are higher than the calculated ones. The alloying elements can also form nitrides or carbides (in case of chromium, molybdenum and vanadium) what results in a higher N and/or C concentration or simply shift the lines to lower nitriding and/or carburizing potentials (in case of carbon) due to the higher base level of carbon (according to figure 4). The total content of nitrogen and carbon increases with increasing alloying elements. In agreement with the calculations in all cases an ϵ -carbonitride-layer was formed.

	ϵ -phase	N [wt.%]	C [wt.%]	Σ (N,C) [wt.%]
calculated	✓	6-7	1-2	7-9
AISI 1015 / Ck15	✓	8.3	2.5	10.8
AISI 1045 / Ck45	✓	7.9	2.6	10.5
AISI 1060 / Ck60	✓	8.6	2.7	11.3
AISI 4140 / 42CrMo4	✓	8.5	2.6	11.1
AISI H13 / X40CrMoV5-1	✓	8.2	3.3	11.5

Table 3: N and C contents of the nitrocarburized steels (570 °C, 4 h, $K_N=1$, $K_{CB}=0.3$)

Further experimental results according to K_N and K_{CB} controlled nitrocarburizing are published in /15, 16/. An expert system is being built up containing all necessary information to predict results of estimated nitriding and nitrocarburizing conditions with the aid of artificial intelligent tools /17/.

8 Summary and Conclusions

Because of the number of factors influencing the nitriding/nitrocarburizing result, a control of the process is necessary for quality management and economic use of process gases. Controlling the potentials during gaseous nitriding or nitrocarburizing is also important for creating defined layers on steel components, e. g. a white layer of γ' - or ϵ -(carbo-)nitride. But in addition to the potentials of nitrogen and carbon the kinetics of layer growth and the steel itself have to be considered, too. This paper shows the current methods for controlling nitriding or/and carburizing potential and the available equipment for the latter:

- To control the layer growth, e. g. in cases where a tiny or even no white layer is desired, the nitrogen transfer speed can be influenced by diluting the reaction atmosphere with nitrogen.

- For controlling the nitriding potential the measurement of either ammonia or hydrogen or a measurement of the dissociation causing i.e. an increase of the total furnace pressure are commonly used ways. Keeping the residual ammonia content at the desired level can be done by adding fresh ammonia, pre-dissociated ammonia or a mixture of both.
- The carburizing potential can be controlled by an oxygen probe or measuring the ratio of CO and CO₂ with an infrared analyzer. The regulating gases for carburizing are CO₂, CO, endogas or a mixture of two, while using a mixture of CO and CO₂ provides the best way.
- To control post-oxidation treatments the oxidation potential can be directly derived from an oxygen probe. The gas atmosphere is controlled by injection of water or NO₂.
- Further modern control variants, where the potentials are not fixed during the process, are phase control and weight percentage control. In these control variants the process parameters K_N and K_C are adjusted automatically throughout the stage aiming for certain white layer properties.

There exist different experimental and theoretical phase diagrams providing a base for the selection of process parameters for nitriding and nitrocarburizing. Alloying elements like chromium, molybdenum, vanadium and even carbon have an influence on phase boundaries and thus have to be considered as well. This means treating real materials presuppose a good experimental experience or a modification of ternary Fe-N-C system to alloys, which has already been achieved for some un- and low-alloyed steels but there is still a big need for further research.

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