Investigation of diffusion behavior of carburized sheet metal in hot stamping

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Abstract. Today's manufacturing of structural car body parts faces several challenges, like forming accuracy and passenger safety. Besides these two requirements, lightweight design plays an important role. One possibility to fulfill these partially rivalling demands is the application of hot stamped parts. The combination of hot forming and in die quenching reduces not only springback, but also results in tensile strengths of up to 1500 MPa. This makes a simultaneous reduction of sheet thickness and therefore weight reduction possible. Further development enabled a tailored adjustment of mechanical properties, for example by applying different cooling conditions along the parts. One of the biggest issues of these state of the art processes is the formation of a transition zone due to heat transfer. A promising approach to adjust the mechanical properties with a minimized transition zone is the carburization of sheet metal. Therefore, the parts are coated with graphite, heat treated and subsequently quenched. In this work, the time variant process of carbon diffusion is investigated. Sheets with two different thicknesses are carburized and quenched. The resulting mechanical properties are analyzed using uniaxial tensile tests and microhardness measurements. The results are correlated with the carbon content measured by EDX-analysis.

Keywords: Hot stamping, Heat treatment, Carburization

1 Introduction

Ecological aspects as well as increasing safety standards are two reasons for growing importance of lightweight construction in automotive industries [1]. There are different strategies to improve lightweight design in today's manufacturing of structural car body parts. One of them is the substitution of commonly used steel grades by either light-weight materials such as aluminum or materials with higher strength [2]. By using high or highest strength materials, sheet thickness can be reduced, which leads to a weight reduction of the components. Since high strength materials have only limited formability and high process forces are required, temperature assisted forming processes are necessary [3]. Hot stamping of ultra-high strength materials has developed to a state of the art process for manufacturing lightweight, but safety-relevant components.

There are two different process routes in hot stamping. In direct hot stamping, the sheets are austenitized, transferred to the press and then subsequently formed and in-die quenched. Tensile strength of up to 1500 MPa for the commonly used boron manganese steel 22MnB5 can be achieved. These high strengths are a result of a fully martensitic structure which is developed when a cooling rate of at least 27 K/s is ensured during in-die quenching and forming [4]. Contrary to direct hot stamping nearly complete preforming is done before austenitization in the indirect hot stamping process. The parts are quenched and calibrated in the press subsequently to the heat treatment [5]. An overview of the process steps in direct and indirect hot stamping can be seen in Figure 1. Since decarburization and oxidation occur during austenitization, only coated sheet material is used for hot stamping. In direct hot stamping, Al-Si coatings are commonly used [6]. Due to the limited formability of Al-Si at room temperature, other coatings, such as zinc, are preferred in indirect hot forming [4]. Furthermore, Zn-coatings offer cathodic corrosion protection [7].

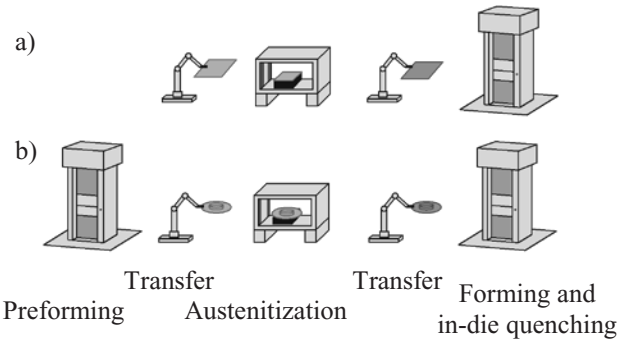

Fig 1. Overview of the process steps in direct (a) and indirect (b) hot stamping according to [8]

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One challenge of hot stamped components is a low residual elongation. Especially in crash relevant structures high energy absorbance is desired. An example for this are b-pillars, where the bottom of the part should have increased ductility for better energy absorbance, while the upper part should retain its structural integrity for passenger safety. By adapting different steps of the hot stamping process, it is possible to manually adjust the resulting mechanical properties. Strategies for tailoring of mechanical properties in hot stamping are partial hot stamping, partial austenitization, annealing and tailored products. By preventing a full austenitization of particular areas, or decreasing the heat transfer between tool and sheet below the critical value, it is possible to retain the original ferritic-pearlitic microstructure besides the martensitic structure [9]. One challenge of these state of the art process variants is the formation of a transition zone due to heat transfer between areas of different temperatures. According to Feuser [10], manufacturing of parts with a transition zone less than 30 mm is not feasible.

A method for minimizing this transition zone, while still being able to adjust the mechanical properties, is tailored carburization [11]. In this process, the sheet metal is locally coated with graphite and subsequently carburized. Areas, where carburization is not desired, are masked with boron-nitride. During the heat treatment in the oven at elevated temperatures, the carbon atoms diffuse into the base material. The locally increased carbon content results in higher strength in these areas after quenching. Existing process understanding from case hardening cannot be transferred directly due to different dimensions and objectives. Since carburization is a time variant process and depends on temperature, relevant process parameters have to be analyzed. Aim of this work is to investigate the influence of sheet thickness and several heat treatment parameters on mechanical properties, such as tensile strength or Vickers hardness and carbon content after carburization. Depending on these influencing factors, a process window for carburization of sheet metal will be identified. The process window identified within this work will be the basis for the application of tailored carburization. Furthermore, the applicability of energy-dispersive X-ray spectroscopy to the analysis of carbon content is investigated.

2 Material and Methodology

A hot rolled, uncoated complex phase steel CP-W® 800 is used in this work. The investigated sheet thicknesses are $t_{01} = 1.6$ mm and $t_{02} = 2.5$ mm. Compared to conventional hot stamping steel 22MnB5, CP-W® 800 has a lower carbon content. The difference between both alloys is 0.11 wt%. This enables the possibility of further carburization. The chemical composition of CP-W® 800 can be seen in Table 1.

The carbon source for carburization is a graphite foil of type 90/10 from KERAFOL Keramische Folien GmbH with a thickness of 0.15 mm. The bonding between base material and graphite is realized by application of the adhesive COT Resbond 931C (Polytec PT GmbH). During air curing, the ceramic binder evaporates, so that the adhesive consists of 99% graphite after hardening. To limit oxidation and decarburization during austenitization, the specimens are wrapped into heat treatment foil with a thickness of 0.051 mm from Schröder Industrieöfen GmbH.

Table 1. Chemical composition of CP-W® 800 in wt% [12]

C max.	**Si max.**	**Mn max.**	**P max.**	**S max.**
0.14	1.00	2.20	0.080	0.015
Al total	**Ti + Nb max.**	**Cr + Mo max.**	**V max.**	**B max.**
0.015 – 2.0	0.25	1.00	0.20	0.005

Besides the thickness of the steel sheets, heat treatment temperature and holding time are investigated as influencing factors. The different specifications are shown below in Table 2. Subsequently to the carburization in the oven, the specimens are quenched in water.

Table 2. Investigated influencing factors

Investigated parameter	**Variation**
Sheet thickness t_0 in mm	1.6, 2.5
Heat treatment temperature T_{oven} in °C	900, 950
Holding time t_{hold} in min	5, 60, 180, 360

After carburization and quenching, tensile tests are conducted using a universal testing machine Zwick Z100 in combination with a digital image correlation system Aramis (GOM GmbH). Afterwards, small segments are separated from the tested samples and embedded in epoxy resin for microhardness measurements in a Fischerscope HM1000.

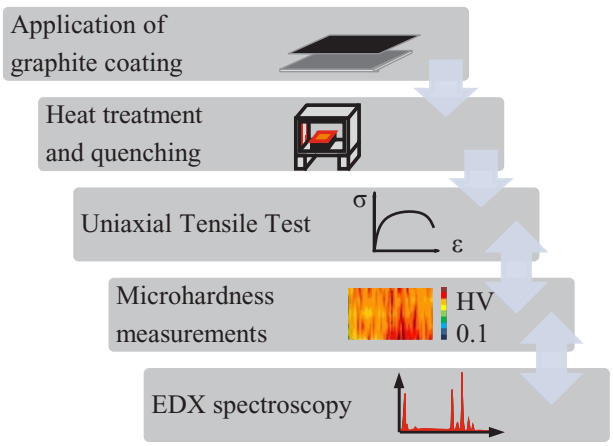

Fig. 2. Used methodology in this work.

The Vickers hardness HV 0.1 is measured on the middle plane of the samples' cross section as well as along the sheet thickness. Afterwards, carbon content of

the carburized samples is measured via energy dispersive X-ray spectroscopy (EDX). The measurements are carried out with a scanning electron microscope of type ZEISS MERLIN GEMINI II, which is equipped with an OXFORD X-MaxN Silicon Drift Detector 50 for EDX analysis. The results are correlated with mechanical properties from tensile tests and microhardness measurements. An overview of the used methodology is given in Figure 2.

3 Results

3.1 Tensile strength

Figures 3 and 4 show the resulting tensile strength after carburization at 900 °C and 950°C in comparison with uncoated specimens. It can be seen in Figure 3, that an increase in tensile strength by carburization is achievable. After a heat treatment of five minutes at 900 °C, the values are below the initial state, with the best results for a sheet thickness of 1.6 mm. After one hour, the tensile strength of the specimens with a thickness of 1.6 mm is slightly increased to (787.2 ± 4.9) MPa. For the samples with t_{02} = 2.5 mm, the values are decreased, independent of potential carburization. After three hours of heat treatment, major differences can be seen. While tensile strength is further decreased for specimens without graphite coating, carburization results in a significant increase in tensile strength. The maximum of tensile strength of (1258.9 ± 2.0) MPa is reached for specimens with a sheet thickness of 1.6 mm. This is significantly higher compared to the initial state with (840.4 ± 1.0) MPa and the specimens with t_{02} = 2.5 mm. Doubling the dwell time to six hours results in a decrease of tensile strength of carburized samples to (855.4 ± 23.5) MPa for t_{01} = 1.6 mm and (809.9 ± 21.8) MPa for t_{02} = 2.5 mm, respectively. This can be explained by decarburization and continuous grain growth as simultaneous effects to carburization. The tensile strength of specimens without graphite coating remains on the same level as before.

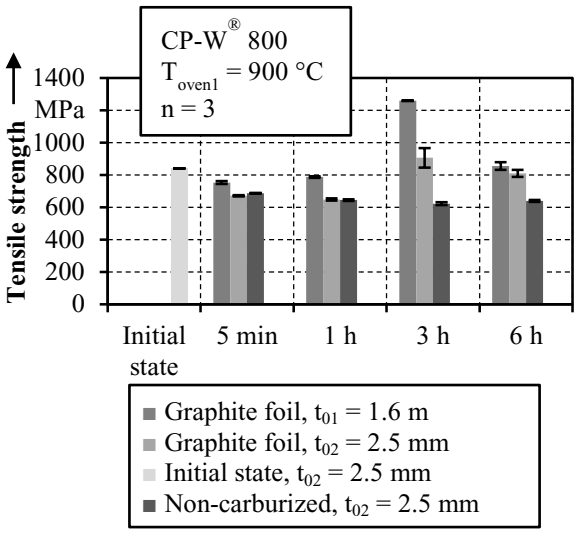

Fig. 3. Tensile strength of coated and uncoated specimens after a heat treatment at 900 °C

Figure 4 shows the resulting tensile strength after a heat treatment at 950 °C and subsequent quenching. The trend of tensile strength is similar to Figure 3, with a difference only after five minutes of heat treatment. Contrary to Figure 3, the carburized samples with a sheet thickness of 2.5 mm show the highest tensile strength with (726.7 ± 25.0) MPa. Considering the standard devitiation, the difference to samples with a thickness of 1.6 mm is not significant. Except for the samples heat treated for five minutes at 900 °C, the investigated specimens with a sheet thickness of 1.6 mm achieve higher values of tensile strength compared to those with a thickness of 2.5 mm. With decreased sheet thickness, the diffusion path of carbon atoms is decreased as well. As a result, the strength gradient over the sheet thickness is smaller compared to t_{02} = 2.5 mm. Therefore, overall tensile strength of the samples is increased.

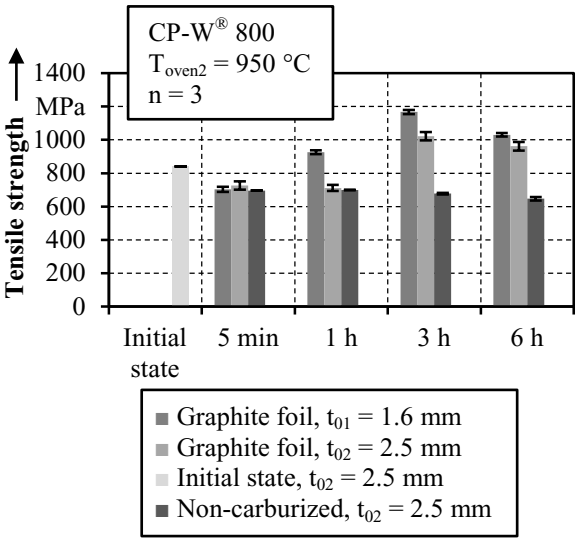

Fig. 4. Tensile strength of coated and uncoated specimens after a heat treatment at 950 °C

Comparing the absolute values of tensile strength in Figure 3 and Figure 4, further differences are visible. It can be seen, that a heat treatment at 950 °C results in higher tensile strength than a heat treatment at 900 °C. An exception can be seen for the maximum in Figure 4 with (1167.2 ± 12.8) MPa for three hours and t_{01} = 1.6 mm. This is about 90 MPa lower compared to the maximum in Figure 3. Basically, carburization is improved at higher temperatures. This explains the elevated values for tensile strength after a heat treatment at 950 °C. Furthermore, not only carburization, but also decarburization is reinforced when temperature is increased. Parallel ongoing decarburization and grain growth are an explanation that the overall maximum tensile strength is reached at a heat treatment temperature of 900 °C instead of 950 °C.

3.2 Vickers hardness

Figure 5 shows the Vickers hardness HV 0.1 after carburization between five minutes and six hours at 900 °C and 950 °C in comparison with the initial state before heat treatment. The measurements are conducted

in the middle plane of the samples' cross section. Furthermore, the distribution of Vickers hardness HV 0.1 along the sheet thickness is shown in Figure 7 for selected samples.

Figure 5 corresponds well with the findings from tensile testing. Carburization shorter than three hours results in lower Vickers hardness compared to the initial state with (353.4 ± 2.1) HV 0.1. The only exception is a heat treatment of one hour at 950 °C, as already seen in Figure 4. For a sheet thickness of 2.5 mm, a significant improvement of Vickers hardness up to (406.2 ± 9.4) HV 0.1 can be achieved only for six hours of carburization at a temperature of 950 °C. Again, the best results can be seen for a sheet thickness of 1.6 mm. The maximum is (474.4 ± 4.2) HV 0.1 for carburization parameters of three hours and 950 °C. After six hours, the hardness is only around 8.5 HV 0.1 below the maximum. For 900 °C, the difference between three and six hours is 24.6 HV 0.1 with (450.4 ± 3.6) HV 0.1 as the higher value after three hours. Contrary to Figure 3 and Figure 4, the overall maximum is achieved for three hours at 950 °C instead of 900 °C.

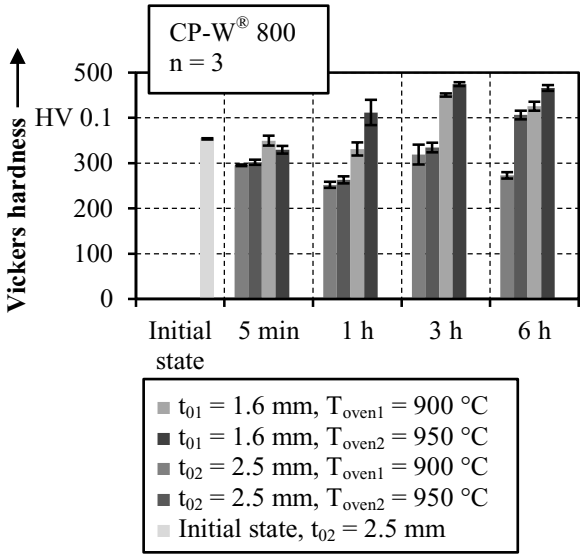

Fig. 5. Vickers hardness of carburized samples in comparison with initial state

For further investigation, the distribution of Vickers hardness along the sheet thickness is analyzed. The location of the measuring points is shown in Figure 6, while the resulting values can be seen in Figure 7. The distance between the measuring points is (97.8 ± 0.4) µm for samples with a thickness of 1.6 mm and (112.0 ± 0.02) µm for specimens with a thickness of 2.5 mm.

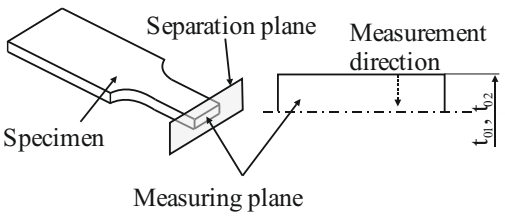

Fig. 6. Location of measuring points for the distribution of Vickers hardness along the sheet thickness.

Figure 7 shows the influence of carburization time. After three hours at 950 °C, an almost homogeneous hardness distribution with values ranging from (459.6 ± 0.04) HV 0.1 to (472.7 ± 9.5) HV 0.1 can be seen. As already mentioned, decarburization and grain growth are two parallel ongoing effects. This is shown for samples with a heat treatment time of six hours. The hardness is decreased to (366.6 ± 6.6) HV 0.1 for a thickness of 1.6 mm and (356.0 ± 7.8) HV 0.1 for samples with t_{02} = 2.5 mm, respectively. Vickers hardness increases with growing distance from the surface. From the surface to a distance of around 0.2 mm, samples carburized for six hours at 950 °C show high accordance in their hardness, independent of the sheet thickness. From there to the mid-section, increased Vickers hardness can be seen for samples with a thickness of 1.6 mm. Due to the lower thickness, diffusion of carbon has proceeded into deeper regions of the material with regard to the overall thickness. Furthermore, the distribution of hardness is more homogeneous in the sheet plane as well. This can be seen by increased standard deviation for specimens with a thickness of 2.5 mm.

Figure 7 also shows an explanation for the different mechanical properties for specimens with carburization temperatures of 900 and 950 °C after a holding time of three hours. As already mentioned, after carburization of three hours at 950 °C, a very homogenous hardness distribution is achieved. Contrary to that, a heat treatment temperature of 900 °C results in a clear hardness gradient. In the middle of these samples, Vickers hardness is around 12.6 to 20.2 HV 0.1 below the values of specimens with a heat treatment temperature of 950 °C. This can be explained with a decreased diffusion coefficient due to lower temperature. Apart from the midsection, samples carburized at 900 °C show up to 82.3 HV 0.1 higher values for Vickers hardness. Besides decarburization, which is especially present in the surface near region, grain growth due to elevated temperatures is a possible explanation [13], which starts around 950 °C [14].

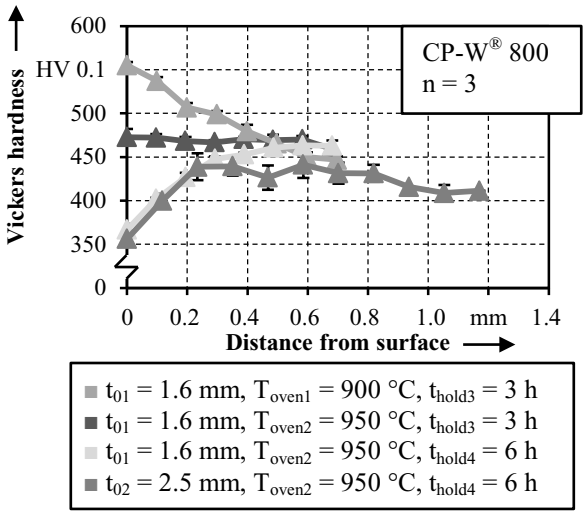

Fig 7. Distribution of Vickers hardness along the sheet thickness of selected samples with a sheet thickness of 1.6 and 2.5 mm after carburization of three and six hours at 900 and 950 °C

Calculating the average Vickers hardness along the sheet thickness, highest values are achieved for sheets with a thickness of 1.6 mm and carburization of three hours at 900 °C with (492.4 ± 19.1) HV 0.1, followed by three hours at 950 °C with (468.7 ± 5.3) HV 0.1 and six hours at 950 °C with (435.4 ± 16.9) HV 0.1. The lowest average Vickers hardness along the sheet thickness can be seen for samples with a thickness of 2.5 mm and heat treatment parameters of six hours and 950 °C with (419.5 ± 16.2) HV 0.1. This corresponds well with the results from tensile testing.

3.3 EDX analysis

Figures 8 and 9 show the carbon content of four different samples measured with EDX spectroscopy. The carbon content of samples carburized at 900 °C is illustrated in Figure 8, while the measurement data for specimens heat treated at 950 °C can be seen in Figure 9. The values were measured via line measurement along the sheet thickness. For the presentation of the results the measured values were averaged over a range of 100 µm. This was done near the surface and in the mid-section of the samples.

The carbon content of samples with a sheet thickness of 1.6 mm carburized for three hours at 900 °C is depicted in Figure 8. The results are similar to the results from Vickers hardness measurements. In the mid-section, the carbon content is lower compared to the values in the surface-near area of the specimens. Considering the standard deviation, the effect is not significant. In addition to the statistical error, displayed by the standard deviation, the measurement data contains an overall absolute measurement error due to low accuracy of energy-dispersive X-ray spectroscopy on light elements such as carbon [15]. Therefore, only a qualitative analysis of carbon content is done within this work.

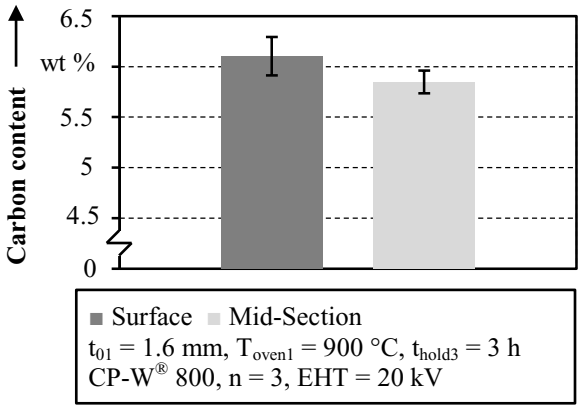

Fig 8. Carbon content of sample with a sheet thickness of 1.6 mm carburized for three hours at 900 °C measured via EDX spectroscopy

The carbon content of samples carburized at 950 °C can be seen in Figure 9. Regarding the specimens with a holding time of three hours and a thickness of 1.6 mm, a very homogeneous distribution of carbon content can be seen. There are only small differences between the mid-section and near-surface areas. A comparable behavior was already shown for the distribution of Vickers hardness HV 0.1 in Figure 7. The standard deviation in the mid-section is increased compared to the surface-near area, which might be a sign for inhomogeneous carburization along the sheet plane. Since possible contaminations of the vacuum chamber have an influence on the measurements, a comparison of the results of EDX spectroscopy amongst each other is not possible.

The results for samples with a thickness of 2.5 mm carburized for six hours at 950 °C correspond well with the findings from Figure 7. Comparing the carbon content in the surface-near area to the values in the mid-section, a clear difference can be seen. The weight percentage of carbon in the mid-section is significantly higher in comparison with the weight percentage of carbon near the surface. This supports the assumption, that decarburization takes place simultaneously to the desired carburization process.

The carbon content of samples with a sheet thickness of 1.6 mm and carburization parameters of six hours and 950 °C can be seen in Figure 9, too. The results are contrary to the findings in Figure 7. The Vickers hardness measurements showed that the hardness is decreased near the surface. Decarburization was stated as possible reason for the gradient. The carbon content measured via energy-dispersive X-ray spectroscopy shows a different behavior. The weight percentage of carbon is increased in the surface-near area and lower in the mid-section of the samples.

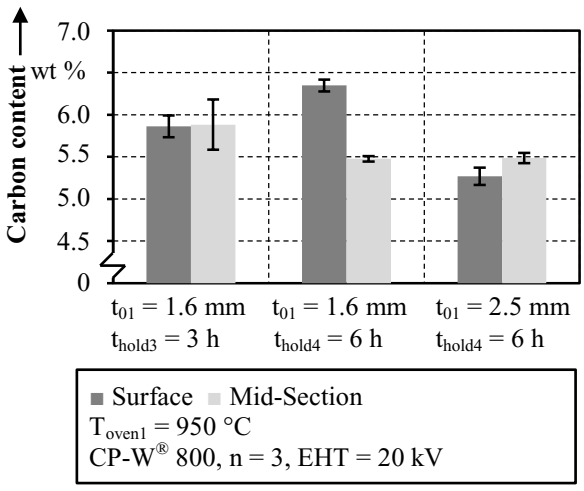

Fig 9. Carbon content of samples carburized at 950 °C measured via EDX spectroscopy

On basis of the investigations carried out within this work, EDX is not applicable for neither quantitative nor qualitative analysis of carbon content. In order to verify the findings in Figures 8 and 9, or as a possible alternative with increased accuracy, wavelength-dispersive X-ray spectroscopy (WDX) has to be taken into consideration. Since WDX spectroscopy faces the same challenges in measuring light elements such as carbon, further investigations have to be carried out as well.

4 Conclusion

Within this work, different carburization parameters such as temperature and holding time as well as the influence of sheet thickness have been investigated. Therefore, the samples were coated with graphite foil, heat treated and subsequently quenched. It was shown, that an improvement of mechanical properties of sheet metal is achievable by carburization. The tensile tests indicated, that sheet thickness has an significant influence on mechanical properties after carburization. Samples with a thickness of 1.6 mm showed higher tensile strengths compared to specimens with a thickness of 2.5 mm. Furthermore, it was revealed, that negative effects such as grain growth and decarburization have an influence as well. This was especially apparent for samples carburized longer than three hours. Since temperature and dwell time have a positive as well as a negative effect on the resulting mechanical properties, the process window is limited to three hours for both investigated temperatures. An extended process window includes carburization times of up to six hours for a temperature of 950 °C. This process window is applicable for both investigated sheet thicknesses. Considering an industrial application of the process, the identified process window is not feasible for production lines with cycle times of few seconds, but is an alternative for more flexible productions of high strength crash relevant parts.

The qualitative results from EDX spectroscopy were partly in accordance to the findings from other investigations such as Vickers hardness. Taking into account an overall error due to the limitations of the measurement method, a quantitative analysis of carbon content was not possible. As a possible improvement in the determination of carbon content, the application of wavelength-dispersive X-ray spectroscopy has to be investigated. Although it offers increased accuracy, WDX faces the same challenges in measuring light elements.

Since an applicable process window was identified within this work, further research has to focus on the detailed investigation of the transition zone between carburized and non-carburized areas.

The authors would like to thank the German Research Foundation (DFG) for funding the project “Tailored Carburization: Gradierung mechanischer Eigenschaften in der Blechebene und über die Blechdicke” (DFG ME 2043/51-1). Moreover, the authors acknowledge the investigations of Yang Yang within the framework of his master thesis at the Institute of Manufacturing Technology (LFT).

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