

## Prevention of scaling by means of recycled process waste gases

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**Keywords:** Retrofit, NoCarb, Hot Forming

**Abstract.** During hot forging of steel materials, the blanks are subjected to various heating processes. During these processes, scale is formed, which can lead to a mass loss of up to 3%. The additional mass required to compensate this material loss for a given forging component has a significant impact on the process emissions, as the production of the billet material has the highest impact on the overall CO<sub>2</sub> footprint of metal forming products [1]. Additionally, descaling operations such as upsetting are required to guarantee forging quality and process stability. At the same time, large quantities of process waste gas are emitted in the production of raw materials and components. These burnt gases have lower oxygen concentration due to the prior chemical combustion reaction. This work addresses the question, whether these burnt gases can be utilized as a forging process atmosphere. This would not only reduce material loss, but would also result in a reuse of the process waste gas. In order to retrofit existing forging infrastructure, a tooling system with a gas-tight enclosure was constructed and realized in a forming press. Defined gas combinations were fed into the enclosure to create an oxygen-reduced atmosphere. First, different gas combinations were investigated in annealing tests. The three most promising ones were then selected for the forging tests. The enclosure contained a heating, transport, forming and collecting unit. The blanks were fed in through a magazine and inductively heated to 1200 °C, formed and cooled under the defined atmosphere. In each atmosphere, 100 components were forged from the material 42CrMo4. Furthermore, it was investigated whether forming under a gas atmosphere has an influence on tool wear as scale can act as an abrasive. The investigations showed that both the surface of the starting material and the oxygen concentration of the atmosphere have a significant influence on scale formation. The amount of scale formed was reduced by up to 74% compared to an oxygen atmosphere. The adhesive layer on the upper dies was reduced with decreasing oxygen concentration. On the lower dies was an increased adhesive build-up.

### Introduction

Considering current findings on climate change, resource efficiency and the reduction of CO<sub>2</sub> emissions are becoming increasingly important both politically and socially. Measures that contribute to climate and environmental protection are therefore of great interest in all fields [2]. In addition to purely economic parameters, these criteria are increasingly evolving into competitive factor in the business to business environment as well as in the end consumer market [3]. The EU Commission has proclaimed the vision of a climate-neutral economy by 2050 [4]. One of the largest emitters of greenhouse gases is the manufacturing industry. Large quantities of CO<sub>2</sub> are produced both during the manufacture of construction materials and the production of goods from these materials [5] as well as during the recycling of goods at the end of their life cycle [6]. The greatest saving potential is undoubtedly achieved by reducing the resources used, e. g. through a higher material efficiency factor (MEE). This is because only the materials that are actually needed

for a product should be produced. Further potential savings lie in lightweight construction and materials, the use of efficient, fully digitalised and automated production processes and the development of suitable reuse concepts, according to the cradle-to-cradle principle [7].

During hot forging of steel materials, oxidation of the raw parts results in pronounced scale formation, which leads to a significant loss of material (approx. 2% to 3% of the mass used) [8]. The scaled material is therefore no longer available for the manufacture of formed products and reduces the usable mass after forming.

The aim of these investigations is to carry out hot forging in an oxygen-reduced atmosphere in order to suppress the formation of scale. The study examines the use of low-oxygen, industrially emitted exhaust gases in an existing forming process. By reducing mass losses, the CO<sub>2</sub> footprint of the components produced can be lowered.

Scale or iron oxide is much harder and more brittle than steel and therefore acts as an abrasive in the tribological contact zone between die and billet during forging. It will also be investigated if lower quantities of scale also mean less abrasive wear on the tools and increase the service life of the tool. As the tool itself also contributes to the CO<sub>2</sub> footprint, its impact can be reduced by using it for an extended period and replacing it less often. By generally avoiding scale, upstream processes such as the upsetting for descaling or sand-blasting can also be completely removed from the process chain.

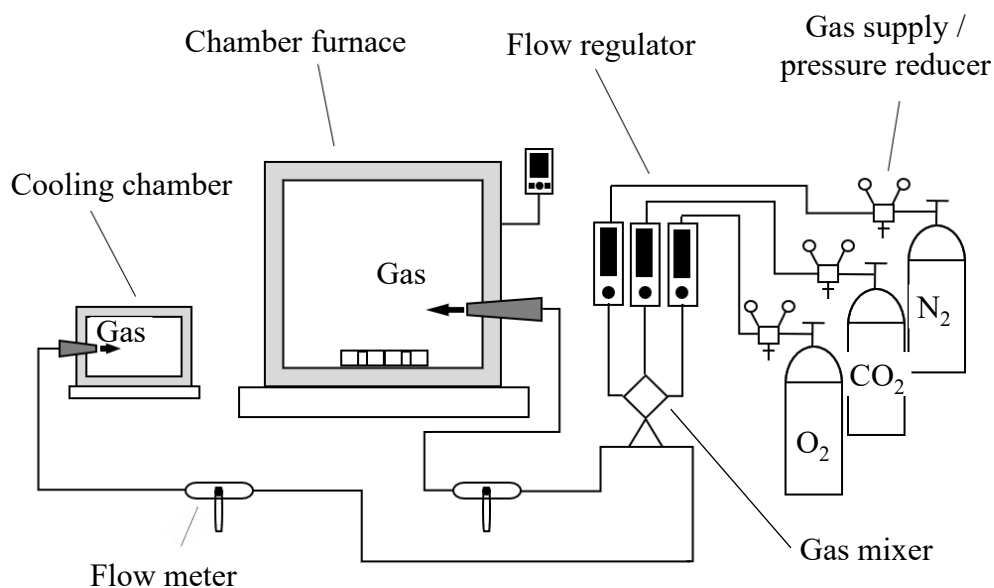
### Material and Methods

To determine the influence of the gas combinations on the scaling behaviour, a tempering test was carried out. To determine the difference in weight before and after annealing, the samples were weighed prior to and after the process. The scaling behaviour was quantified as a factor of the atmosphere by the weight loss. The samples were annealed in a sample holder in a previously purged inert gas furnace for 60 min at 1100 °C in the selected atmosphere. This temperature was chosen as it is a common hot forming temperature for steel [9]. This holding time in the furnace was significantly longer than in conventional industrial forging processes. It served to qualitatively assess the potential and pre-select the different gas compositions. After this time, the samples were transferred from the inert gas furnace to a cooling chamber, which was also purged with the same gas composition as the furnace. The sample holder was transported manually with a transfer time of approx. 5-8 s and came into contact with the ambient air during the transfer. The samples remained there until their temperature had fallen below 500 °C, as no significant scale formation takes place below this temperature [10, 11]. Depending on the process, process waste gases can have a wide variety of compositions. For this purpose, Huczkowski et al. simulated the compositions of flue gases produced during the technical combustion of fuels [12]. These are mainly composed of nitrogen (N<sub>2</sub>), carbon dioxide (CO<sub>2</sub>), water (H<sub>2</sub>O), oxygen (O<sub>2</sub>) and sulphur dioxide (SO<sub>2</sub>). Lee and Choi modeled the waste gas composition of a heating process with the combination N<sub>2</sub>+CO<sub>2</sub> (10%) + O<sub>2</sub> (5%) [13]. Gas combinations with different proportions of N<sub>2</sub>, CO<sub>2</sub> and O<sub>2</sub> were selected to investigate the influence of different combustion processes. Nitrogen was used as the main gas and the proportions of carbon dioxide and oxygen were varied. A pure nitrogen atmosphere was also selected, which used in hardening or tempering processes, for example. Environmental statements from large hardening plants such as Reese (Härterei Reese Weimar GmbH & Co. KG, Weimar) or the BMW Group (BMW Group, Werk Dingolfing) show that these gases are emitted from the processes in significant quantities. The selected combinations are summarised in Table 1. Sulphur dioxide (SO<sub>2</sub>) was not added as it represents a very small proportion in the sources used. Water (H<sub>2</sub>O) would be separated during the preparation and cooling of the gases for the process, which is why it was also not added separately.

*Table 1: Gas compositions used in annealing tests*

Test series	Gas composition
A	Air atmosphere
B	N <sub>2</sub> +CO <sub>2</sub> (5%)+O <sub>2</sub> (10%)
C	N <sub>2</sub> +O <sub>2</sub> (10%)
D	N <sub>2</sub> +CO <sub>2</sub> (10%)+O <sub>2</sub> (5%)
E	N <sub>2</sub> +CO <sub>2</sub> (5%)+O <sub>2</sub> (5%)
F	N <sub>2</sub> +O <sub>2</sub> (5%)
G	N <sub>2</sub> +CO <sub>2</sub> (10%)
H	N <sub>2</sub> +CO <sub>2</sub> (5%)
I	N <sub>2</sub>

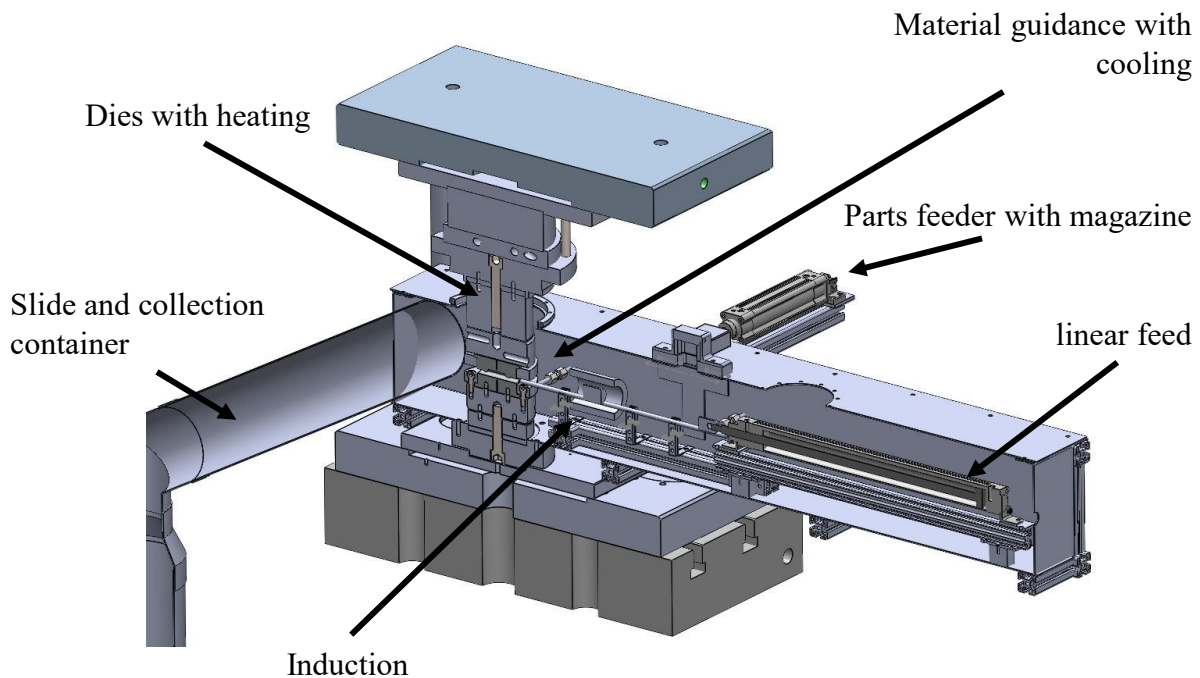
For the tests, the material 42CrMo4 was selected in the variations bright (machined surface) and black (rolled) in order to simultaneously investigate the influence of the surface condition of the blanks on scaling. All cylindrical blanks for these tests had a diameter of 30 mm and a height of 40 mm. For the gas atmosphere, the aforementioned gas combinations of technical gases with corresponding purity were selected. In order to obtain a comprehensive picture of scaling, 24 samples were used for scaling tests in each material and gas combination. A gas mixing module was used to achieve the gas compositions. In this module, the quantity of each gas used could be set using digital flow rate regulators. The gases were then combined and fed into an inert gas furnace and a cooling chamber. By combining the gases before they were actually used, they were able to mix and thus minimise the separation of the gases in the different areas of the oven. A schematic representation of the gas flow displayed Fig. 1.



*Figure 1: Schematic illustration of the gas flow*

For further tests, three gas combinations were selected from the results. On the one hand, the variation with the lowest and the highest scale formation, as well as one a medium-level to verify the process. The surface variation with the stronger scale formation was used to amplify the influence of the gas for analytical purpose and focus on application scenarios with the most potential for improvement.

To carry out series forging tests under defined atmospheres, a tool system was developed which had an enclosure, a collecting container, a heating unit, a linear feed and dies adapted for the tests (Fig. 2). This tool was used in the Lasco SPR 500 screw press (LASCO Umformtechnik GmbH, Coburg) with a nominal force capacity of 5000 kN. A magazine, which feeds the components into the tool, allows continuous forging series to be carried out. In order to reproduce long-term behaviour under statistically verified conditions, forging series of 100 components per atmosphere were carried out. New dies were used for each forging series in order to be able to analyse the effects on die wear. The atmospheres were monitored using a lambda probe to check the oxygen content. Before starting the test series, the dies were preheated to 200 °C, as the basic tool temperature is normally between 100 °C and 250 °C [9]. As in the tempering test, the cylindrical blanks had a diameter of 30 mm and a height of 40 mm. They were heated to approx. 1200 °C using the integrated heating unit. In order to avoid contamination of the atmosphere, the tests were carried out under slight overpressure in the enclosure. A reference forging series in an air atmosphere was also carried out in addition to the three selected gas combinations.



*Figure 2: Tool system and housing*

The samples were weighed before the test series were carried out. The blanks were then fed into the housing via a magazine and separated. The samples were pushed into the heating unit by the linear feed. After heating, the samples were pushed into the forging dies and the forming process was carried out. After forming, the samples were pushed onto a chute and collected in a cooled container. On average, the cycle time was approx. 40 s. The atmosphere in the collecting container was the same as in the process. The forged semi-finished products cooled down in the container and were then removed. As in the annealing tests, the scale was removed from the samples by subsequent blasting with glass beads of 90 – 150 µm. The samples were cleaned in

batches of 100 parts from the same series. The removal of non-scaled material was not considered significant due to the selected blasting medium [14]. The weight difference and thus the scale mass loss was determined by comparing the weight of the cleaned semi-finished products with the initial weight. The average material loss is calculated for each test series.

To evaluate the influence on the dies, the surfaces were scanned before and after the test series using the Keyence VR-3200 profilometer with a 12x magnification resulting in macroscopic and 3D images. The comparison of the 3D contours before and after the forging test series enables the overall changes in the tool surface structure resulting from the tribological conditions.

**Results**

The weight loss in the tempering tests for the different process gas mixtures is displayed in Fig. 3 shows that weight loss decreases when oxygen and carbon dioxide content is reduced. In addition, the blank samples show a significantly lower weight loss than the black samples. A reduction in weight loss of over 50% could be determined for both the black and the blank samples. The relative weight loss in an air atmosphere was 0.5% for the blank samples and 0.6% for the black samples. This relative loss could be reduced to 0.2% for bare samples and 0.3% for black samples under pure nitrogen. The weight of the black samples before forming was 224.36 g with a standard deviation of 0.37 g, the weight of the blank samples 222.91 g with a standard deviation of 0.19 g. The standard deviation of the values shown in Fig. 3 varied depending on the surface and the gas composition used. It was between 0.30 g and 1 g for black surfaces and between 0.27 g and 0.51 g for blank surfaces.

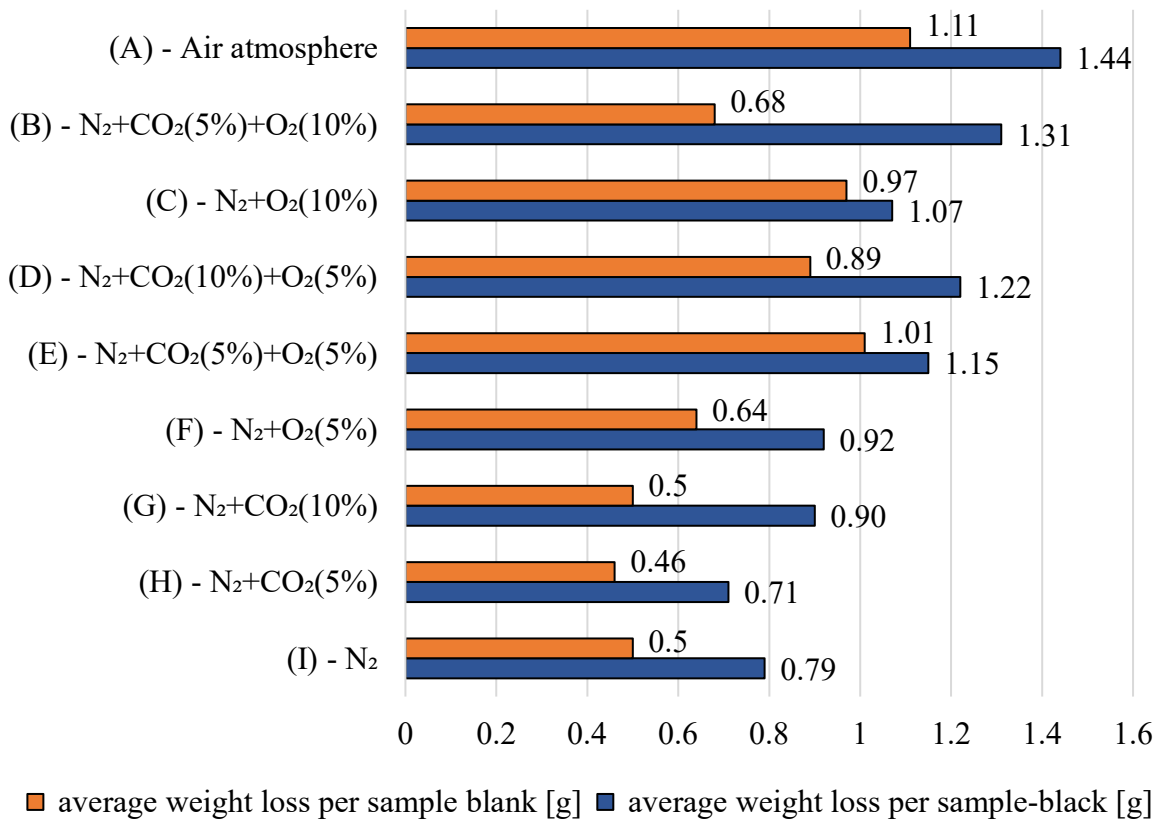


Figure 3: Mass loss due to scale in annealing tests of 42CrMo4

In the subsequent series of forging tests under the gas atmospheres A (air atmosphere), E (N<sub>2</sub>+CO<sub>2</sub> (10%)+O<sub>2</sub> (5%)), H (N<sub>2</sub>+CO<sub>2</sub> (5%)) and I (N<sub>2</sub>) were selected. A significant reduction in

the loose scale around the tool compared to the reference series was already observed after completion of test series E ( $N_2+CO_2$  (10%)+ $O_2$  (5%)). In test series H ( $N_2+CO_2$  (5%)) and I ( $N_2$ ), virtually no chipped scale pieces could be detected around the die.

The evaluation of the component mass loss is shown in Fig. 4 for each test series. The mass loss decreases with decreasing oxygen and carbon dioxide content. The mass loss in air atmosphere was used as a reference. The relative mass loss decreases under the various atmospheres from over 1.6 g in an air atmosphere to less than 0.5 g in a pure nitrogen atmosphere. In percentage terms, the mass loss drops to approx. 74%. The average weight before forming was 224.52 g with a standard deviation of 3.35 g. After blast cleaning of the components, the mass loss shown in Fig. 4 was determined. The standard deviation of the weighed components was between 2.89 g and 3.28 g, depending on the gas composition.

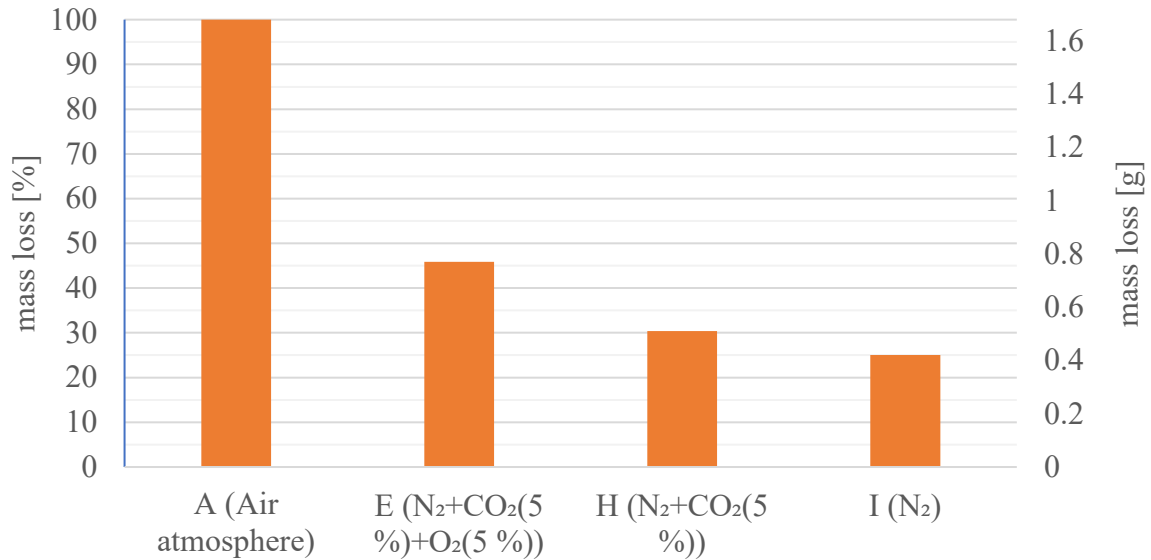


Figure 4: Averaged mass loss of the parts due to scale

Fig. 5 a-d shows the surface of the upper dies. It can be seen that material has been applied to all the dies. This appears to be most evident at die shown in Fig. 5 a. Another conspicuous feature is that there is an increased material adhesion on the ridge path in all the dies. This is particularly evident in Fig. 5 c, area 2. A slight erosion at the edges of this vertical swage channel, which is shown in Fig. 5 a, area 1, can be observed. Minor differences in height can be attributed to measurement inaccuracies. When the contours are superimposed, the reference height is minimally influenced by the average particle size of the spray. Nevertheless, it is observed that the amount of adhesions under the gas atmospheres is lower than in the air atmosphere. A decrease in adhesion can be observed for E ( $N_2+CO_2$ (5%)+ $O_2$ (5%)) and I ( $N_2$ ) compared with the reference with air. It can be seen that a lower oxygen content tends to result in less adhesion on the upper die.

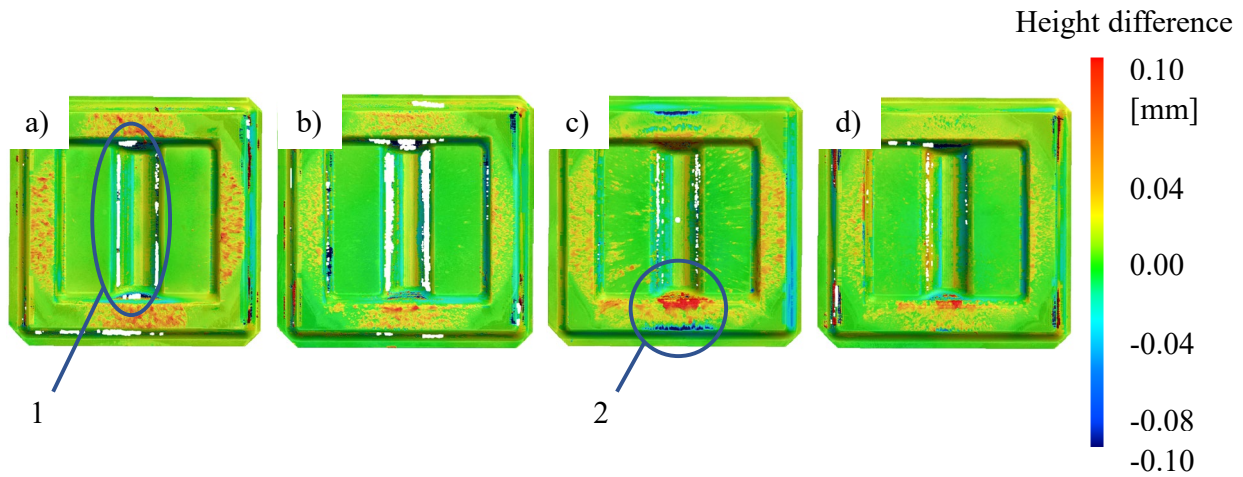


Figure 5: Contour comparison of the upper dies a) Air atmosphere b)  $N_2+CO_2(5\%)+O_2(5\%)$  c)  $N_2+CO_2(5\%)$  d)  $N_2$

In addition to the upper dies, the lower dies were also analysed. These are shown in Fig. 6. It can be seen from these images that adhesion also occurred on all dies. Areas with negative geometric deviations, as shown in Fig. 6 a, area 1, can be detected in all the dies. Compared to the other dies, the die shown in Fig. 6 c shows more significant adhesion. Material build-up in the area of the ridge path on the upper die occurs on all dies. A comparison of the lower dies A (Air atmosphere), E ( $N_2+CO_2(5\%)+O_2(5\%)$ ) and I ( $N_2$ ), as already carried out for the upper dies, did not reveal any reduction in build-up due to the use of a gas atmosphere. However, the areas with negative geometric deviations were reduced.

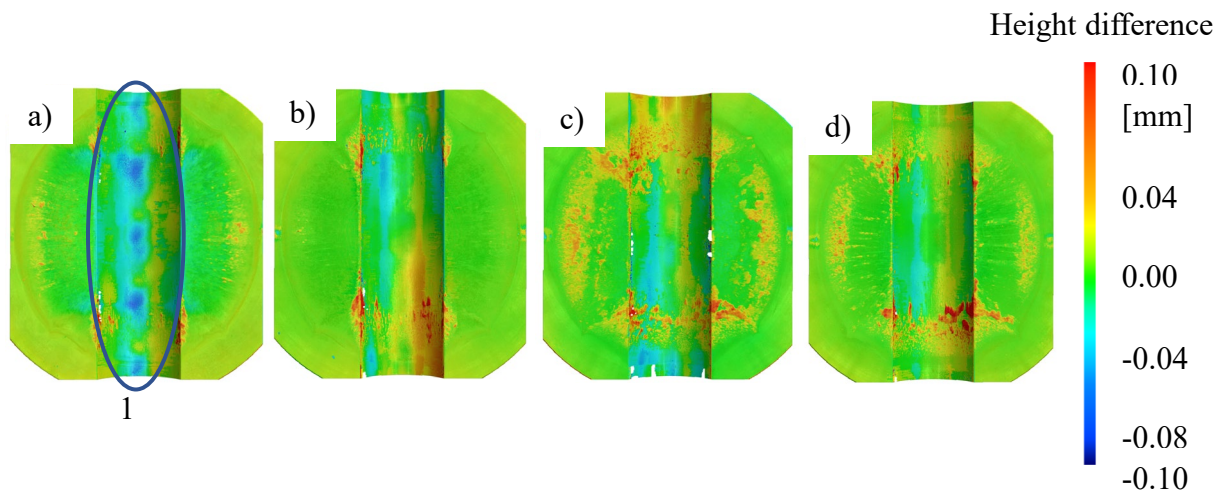


Figure 6: Contour comparison of the lower dies a) Air atmosphere b)  $N_2+CO_2(5\%)+O_2(5\%)$  c)  $N_2+CO_2(5\%)$  d)  $N_2$

### Discussion

A tempering route was modelled using these gas combinations and the material 42CrMo4 in the variations "bright" and "black". The results showed that the reduction of oxygen and carbon dioxide with addition of nitrogen leads to a reduction in the amount of scale formed. The best results were initially achieved under a nitrogen atmosphere with 5%  $CO_2$ . It was expected that a pure nitrogen atmosphere would achieve the best results as no oxygen is available. The results

show, that oxidation is also inhibited under high temperatures when oxygen is bound as CO<sub>2</sub>. Burnt gases with high CO<sub>2</sub> content therefore show high potential as an inert atmosphere in forging. The formation of scale can be reduced by using gases that lower the oxygen concentration in the process atmosphere. Whether the gases have an effect on the forged parts due to diffusion processes or a special atmosphere can be used in the production process must be checked on a case-by-case assessment.

Due to the deviating results, the pure nitrogen and the best atmosphere from the test were selected for further tests. An air atmosphere was also selected as a reference and a gas composition with N<sub>2</sub>+CO<sub>2</sub> (5%)+O<sub>2</sub> (5%). It was found that bare surfaces tend to form less scale. This can be explained by the fact that the "bright" surfaces have a smaller specific surface area due to less roughness. Benefits in terms of reduced material loss in forging "bright" components are however not significant, as the material loss is merely shifted to an earlier stage in the process chain. The peeling process for blank steel rods naturally has high material loss as it is a chip removing method. The removed chips can however be more easily recycled than oxide scale. This method would significantly increase the CO<sub>2</sub> footprint of the component, as the additional process steps and recycling would also have to be included in the overall balance of the component. It is not a reasonable approach to reduce scaling and the CO<sub>2</sub>-footprint and therefore not considered for the subsequent forging tests. For this reason, black samples were used for the further observations.

In subsequent series of tests, in which 100 parts were forged for each gas combination, it was shown that the lowest scale formation occurs under a pure nitrogen atmosphere, as expected. A scale reduction of approx. 1.1 g from the highest to the lowest result was achieved through the series of tests under the controlled atmospheres. This corresponds to a reduction of 74%. It was also shown that a partial reduction of the oxygen content has a significant effect on scale formation. Without the addition of oxygen, a certain amount of oxidation occurs. By reducing the oxygen content to a quarter of the normal level, more than half of the oxidation or the scale formed can be saved. The difference between CO<sub>2</sub> and N<sub>2</sub> is only relatively small. From this it can be deduced that the greatest improvements can be achieved if the residual oxygen concentration is minimized. This can be achieved with N<sub>2</sub> as well as CO<sub>2</sub>.

No significant removal could be recognised in the contour comparisons of the upper dies. However, a clear build-up is recognisable in the area of the burr formation, which decreases as the amount of scale decreases. This can be explained by the fact that the scale forms a thermal protective layer that protects the sample against heat loss to the environment. Due to the absence of scale, it is possible that the components cool down faster and less adhesion occurs, as adhesive effects increase with temperature. The lower dies, on the other hand, exhibit negative geometric deviations in the centre of the dies, which also decrease with decreasing scale formation. This wear may be caused by scale which acts as an abrasive that is pressed into the surface of the die during the forging process. The reduction in abrasive wear was confirmed, which was particularly evident on the lower dies. The reduction in wear is attributed to the lowering of the scale content and the resulting reduction in the quantity of abrasive particles. Contrary to the effects on the upper dies, adhesion increases with decreasing scale. This result is possibly due to the fact that the lower dies surface has higher heat input than the upper dies, due to longer contact time with the billet before and after forging. Effects of increased adhesion with increasing die temperature have already been identified by Lorenz et al. [15]. The increased base temperature and the lack of a scale layer led to increased adhesion. This effect on the lower tool is also expected, since scale acts as an intermediate layer between billet and tool, preventing direct contact and chemical interaction, which can cause adhesion. The thermal and tribochemical effects can be solved by using lubricant. A separate adjustment is necessary as these have been atomised with compressed air up to now. One solution would be to substitute compressed air with pressurised inert or waste gas.



## Conclusions

It has been shown that offgases can be used to reduce scale formation. An influence of the component surface on scale formation could be shown. A scale reduction of up to 74% was achieved, which improves the CO<sub>2</sub> footprint of the components. The abrasive wear on tools was reduced by lowering the amount of scale. As a consequence, tool lifetimes can potentially be increased, further reducing the CO<sub>2</sub> footprint.

## Acknowledgements

This research and development project NoCarb-Forging - ErProFit (Number: 03LB3017) is funded by the Federal Ministry for Economic Affairs and Climate Action (BMWK) within the funding initiative "Technologietransfer-Programm Leichtbau (TTP LB)" (Lightweight construction technology transfer programme) and implemented by the Project Management Jülich (PTJ). The author is responsible for the content of this publication. The authors thank the company "Gebr. Steller KG" for their support in this project.

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