Influence of Process Gas Composition on Laser Cladding Process Characteristics

A. Wank, C. Schmengler, A. Hitzek
GTV Verschleiss-Schutz GmbH, Luckenbach, Germany

W. Kroemmer, M. Runzka, B. Merten
Linde Gas Division, Linde AG, Unterschleissheim, Germany

Abstract
The influence of process gas composition on characteristics of laser cladding processes is studied in detail at the example of a 60 HRC nickel based self fluxing alloy powder. Typically pure nitrogen, argon or helium are used as process gases in laser cladding processes. Besides mixtures of these gases also addition of hydrogen, carbon dioxide and oxygen are applied studying their influence on thermal emission, weld penetration depth and homogeneity, powder usage and crack formation. Use of identical composition of carrier gas and laser process gas is compared to use of different carrier and laser process gases. Oxygen addition increases thermal emission, but does not result in increased weld penetration depth or crack formation tendency. Thereby homogeneity of weld penetration is improved in comparison to use of pure argon. Also, maximum hardness of claddings is achieved when adding oxygen.

Introduction
Applications of laser additive manufacturing technologies including laser cladding have increased rapidly within the last decade due to steadily decreasing prices of laser sources on the one hand and excellent control over heat transfer to powder feedstock and component surface areas in related processes on the other hand. Accordingly laser cladding permits exceptionally low dilution with base materials without compromising metallurgical bonding, small heat affected zones and distortion as well as low porosity and homogeneous, fine grained microstructure of the deposited layers at high reproducibility.

In order to optimize laser cladding processes regarding powder usage and homogeneity of clad material without dependency on the cladding head traverse path extensive research and development has been conducted especially concerning powder feed device design and gas flow control. Initially lateral powder feed using off-axis nozzles has been applied. However, such approach can easily result in asymmetrical weld penetration and a change of welding direction strongly influences shape and microstructure of deposited material [1]. Therefore and because of the typically significantly reduced powder usage off-axis nozzles are mainly used in jobs that do not permit use of coaxial feed nozzles due to geometrical restrictions of the workpieces shape.

Besides annular gap nozzles that generally permit exceptionally small powder jet foci, but are mostly restricted to flat welding position coaxial feed nozzles with typically three to six discrete injectors or injection bores uniting their powder jets on the laser beam axis are most commonly used these days. Both permit powder usage up to 97% in industrial production. CFD has been used as a powerful tool to study the effect of centrally supplied laser process gas, carrier gas and in some cases also externally applied shielding gas flows in order to achieve robust cladding conditions that permit minimum weld seam width and maximum powder usage [2-4]. Thereby the influence of gas flows on particle trajectories with resultant powder focus position as well as particle speed with resultant likeliness of rebounding has been analyzed in the same way like the cooling effect on substrate and melt pool as well as the effect on ambient air entrainment as a consequence of occurring turbulences with according influence on the reactivity of the gaseous environment [5].

While joining by laser welding is often done in keyhole welding mode with according formation of a plasma cloud, laser cladding is carried out exclusively in conduction welding mode. Therefore gases cannot act in a comparable reactive way like in laser nitriding [6-7] or laser oxidation [8] processes that form ceramic layers on top of remelted substrate surfaces. Considerations concerning the choice of type of gas in laser cladding processes have mostly been based on the goal to ascertain sufficiently inert conditions especially in the melt pool area. Therefore primarily argon has been used both as carrier gas and laser process / shielding gas [9]. Nitrogen is mostly applied in order to save gas costs. Helium is only used in a few applications with the major aim to minimize the momentum transfer to powder particles and thereby to achieve particularly low powder particle velocities and minimum deflection of particle trajectories by the laser process gas flow. Typical applications are in the field of titanium alloy component repairs, because component costs justify the use of expensive gas and titanium alloys show low density and according strong susceptibility to momentum transfer.

So far there has been no detailed research on the influence of gas composition including reducing or oxidizing gas contents. This study aims for clarification of the influence of the type of
gases applied in laser cladding processes at the example of a material with relatively low reactivity, i.e. a nickel based self fluxing alloy with nominal hardness of 60 HRC, that is clad on mild steel substrates.

**Experimental**

Experiments are carried out with a high power diode laser type LDF 6000-60 (Laserline GmbH, Mühlheim-Kärlich, Germany) with wavelength between 980 nm and 1,060 nm, a 1,500 µm diameter fiber, 80 mm collimation and 250 mm focal length, which results in 4.7 mm focal spot diameter. Feedstock is fed coaxially using a 6 injection port powder nozzle PN6625 (GTV Verschleiss-Schutz GmbH, Luckenbach, Germany) with 1.5 mm ID injectors or an annular gap powder nozzle Coax8 (Fraunhofer Institute IWS, Dresden, Germany).

A nickel based self fluxing alloy with 60 HRC nominal hardness (GTV 80.15.1, GTV Verschleiss-Schutz GmbH, Luckenbach, Germany) is used as feedstock. The chemical composition determined by ICP-OES is listed in Table 1. In laser scattering analyses using Quantachrome CILAS 920 d₉₀ is measured. The apparent density of the gas atomized powder is 4.3 g/cm³. Flow behavior is characterized by Hall flow testing and a time of 14.7 s/50 g is measured.

**Table 1: Chemical composition of applied powder feedstock**

<table>
<thead>
<tr>
<th>element</th>
<th>content [wt.-%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>nickel</td>
<td>base</td>
</tr>
<tr>
<td>chromium</td>
<td>15.2</td>
</tr>
<tr>
<td>silicon</td>
<td>4.5</td>
</tr>
<tr>
<td>iron</td>
<td>3.8</td>
</tr>
<tr>
<td>boron</td>
<td>3.4</td>
</tr>
<tr>
<td>carbon</td>
<td>0.68</td>
</tr>
<tr>
<td>phosphorous</td>
<td>0.06</td>
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</tbody>
</table>

Milled and degreased S355JR mild steel substrates with 9 mm thickness, 100 mm length and 50 mm width are used.

A first test series concerning the influence of the applied gas composition was carried out using a parameter set that permits production of crack free claddings with PN6625 nozzle using pure argon both as carrier and laser process gas (Table 2).

**Table 2: Process parameter set for basic process gas influence analyses**

<table>
<thead>
<tr>
<th>parameter</th>
<th>value</th>
</tr>
</thead>
<tbody>
<tr>
<td>laser power</td>
<td>2,200 W</td>
</tr>
<tr>
<td>laser process gas flow rate</td>
<td>20 l/min</td>
</tr>
<tr>
<td>carrier gas flow rate</td>
<td>6 l/min</td>
</tr>
<tr>
<td>powder feed rate</td>
<td>7 g/min</td>
</tr>
<tr>
<td>traverse speed</td>
<td>0.25 m/min</td>
</tr>
<tr>
<td>offset / overlap</td>
<td>2.3 mm / 50%</td>
</tr>
<tr>
<td>power density</td>
<td>127.5 W/mm²</td>
</tr>
<tr>
<td>energy input per unit length</td>
<td>5,280 J/cm</td>
</tr>
</tbody>
</table>

Six overlapping seams with 40 mm length have been clad with different process gases. Besides pure argon, helium, nitrogen and carbon dioxide also two-gas mixtures of carbon dioxide with argon, helium, nitrogen and oxygen as well as mixtures of argon and helium with oxygen at volumetric ratio 75/25, 50/50 and 25/75 are used. In case of argon / helium mixtures ratios of 70/30, 50/50 and 30/70 have been applied, because these are standard products. Oxygen and hydrogen could not be used as constituent of the carrier gas for safety reason. Therefore laser process gas is accordingly enriched to result in total gas flow with average oxygen content of 25, 50 and 75%. Also for safety reasons only carbon dioxide / hydrogen mixtures with hydrogen contents of 1%, 3% and 5% are tested.

GTV LPowC system (GTV Verschleiss-Schutz GmbH, Luckenbach, Germany) is used with constant settings for all tests in order to permit comparison of the overall emission of melt pools during cladding processes. Such analyses do not permit determination of the melt pool temperature, but permit a qualitative evaluation of effects on it.

The specimen’s surfaces are checked visually and by USB microscope DNT Digimicro 2.0 concerning crack and scale formation. Powder usage is determined gravimetrically by calculating the ratio of fed powder and the weight gain of specimens.

Metallographic analyses of cross section images include the determination of the weld penetration area and the area of molten material (weld penetration and deposited material combined). The evaluation also includes the determination of the weld penetration depth, the cladding thickness and the depth of heat affected zones with average values and standard deviations.

Microhardness testing HV0.1 and HV1 are carried out in order to determine hardness profiles from the coating over the heat affected zone down to the base material as well as the hardness of different microstructures inside the cladding.

Comparative studies were carried out using an annular gap powder nozzle Coax8. All process parameters were set identical to the use of a GTV PN6625 nozzle except for standoff distance. Instead of 25 mm (the design standoff distance of PN6625 nozzles), Coax8 nozzles show their powder jet focus at the applied distance of 15 mm. Tests were not meant to compare the capability of the powder nozzles, but only in order to check the process gases influence using a powder nozzle that features a different length of particle trajectories on their way to the substrate surface. Besides pure argon also a mixture of argon and 25% oxygen has been used.

A modified powder feeder is applied for comparative studies using oxygen containing carrier gas. First pure oxygen is used both as laser process gas and carrier gas. Then a test series with a total gas composition Ar/25%O₂ is conducted. Two specimens are produced with oxygen only contained in the laser process gas in order to check reproducibility of results.
Additionally an Ar/25%O\textsubscript{2} mixture is applied both as laser process gas and carrier gas for comparison.

**Results**

Average melt pool emission intensity for cladding with different process gases is summarized in Figure 1. The emission differs only a little for use of pure argon, helium, nitrogen or carbon dioxide with carbon dioxide showing a little increased emission intensity compared to the other gases that can be regarded fully inert towards the clad alloy. However, mixtures of carbon dioxide and 25% of argon or helium both result in significantly higher emission intensity compared to use of pure carbon dioxide.

![Figure 1: Melt pool emission measured by LPowC depending on the applied process gas composition](image1)

Oxygen addition to argon, helium or carbon dioxide generally results in strongly increased melt pool emission intensity. Thereby the increase of emission intensity is highest for addition of the first 25% oxygen. However, raise of oxygen content to 50% and 75% still results in further increase of emission intensity.

For addition of hydrogen to carbon dioxide up to a maximum content of 5% there is no obvious influence on melt pool emission intensity.

In 12 out of the 28 produced claddings short cracks are found at the cladding edges (Figure 2). There is no correlation between inertness of process gases and crack formation. Cracks are found in specimens produced with argon/helium mixtures as well as in specimens produced with carbon dioxide and/or oxygen containing process gas.

![Figure 2: Crack formation at the edges of claddings produced with Ar/75%CO\textsubscript{2} (top) and CO\textsubscript{2}/25%O\textsubscript{2} gas mixtures](image2)

Specimen surfaces are shiny for the use of pure inert gases argon, helium and nitrogen including their mixtures. Using process gas mixtures with at least 75% carbon dioxide or any oxygen addition results in the formation of scales. With rising oxygen content in process gas mixtures the surfaces show a more and more intensive green color. Only for use of a H\textsubscript{2}/75%O\textsubscript{2} gas mixture a spalling scale is formed. Therefore, only an incompletely clad specimen that does not permit full comparison is produced.

None of the claddings are completely free of pores. However, pores are all smaller than 20 µm and their content inside the evaluated cross sections is significantly lower than 1%. As individual cross sections do not necessarily provide a representative impression of the pores contained in claddings and because of the generally low porosity, a correlation between gas composition and porosity is not possible with one exemption. Claddings produced with CO\textsubscript{2}/H\textsubscript{2} mixtures show clearly higher porosity, which can be attributed to the formation of water vapor.

Powder usage depending on the applied process gas composition is summarized in Figure 3. Although powder usage only covers a fairly small range between 87% and 96% in total a clear correlation to gas composition is found. Argon/helium mixtures can be considered constant concerning their inert character, but powder usage drops constantly with increasing helium content from 93% to 87%. In the same way...
powder usage drops with increasing helium content in carbon dioxide / helium mixtures. The use of pure helium as carrier gas at the applied flow rate of 6 l/min does not permit a complete transport of powder from the feeder disk into the powder feed hoses, but a small amount of powder remains inside the feeder disk groove. So, the actual feed rate through the powder nozzle is reduced.

Carbon dioxide permits maximum powder usage among the tested pure gases, i.e. 94%. But, also mixtures consisting of carbon dioxide and nitrogen or argon permit roughly the same high powder usage. In contrast the powder usage drops with an increasing content of oxygen in mixtures with argon, helium or carbon dioxide. Also, even small contents of hydrogen in mixtures with carbon dioxide cause a significantly reduced powder usage compared to the use of pure carbon dioxide.

The evaluation of areas representing material deposited on substrates mostly confirms the results of powder usage analyses (Figure 4). Among the pure gases carbon dioxide produces the largest build-ups and deposited material decreases with a rising content of helium in mixtures with argon or carbon dioxide. Also, deposited material decreases with an increasing content of oxygen or hydrogen in the applied two-gas mixtures.

In contrast weld penetration areas of cross section images are particularly large for the use of carbon dioxide rich process gas mixtures, while oxygen addition to argon and helium up to a maximum content of 50% results in particularly small weld penetration areas (Figure 4). Also, mixtures of oxygen and carbon dioxide produce much smaller weld penetration areas than pure carbon dioxide. Only mixtures of carbon dioxide with nitrogen or hydrogen produce even larger weld penetration areas compared to pure carbon dioxide. Besides oxygen also helium has an effect reducing weld penetration areas. A mixture of 50% helium and 50% oxygen produces the smallest weld penetration areas of all specimens (Figure 5).

**Figure 3: Gravimetric powder usage depending on the applied process gas composition**

**Figure 4: Cross section areas representing the deposited material (red) and the weld penetration (blue) depending on the applied process gas composition**

**Figure 5: Cross section images of claddings produced with pure argon (top) and a He/50%O2 gas mixture (bottom) including results of clad material, weld penetration and depth of heat affected zones measurements**
Figure 6 comprises microhardness testing results of two different microstructures inside the produced claddings. Claddings are produced with an overlap of 50%. Accordingly a part of the previously deposited material is remelted and becomes part of the later deposited seam. So, clad material in the vicinity of remelted cladding material is subject to heat transfer from the last deposited seam with an according effect on the local microstructure. Material that is subject to such treatment shows a transition towards an equiaxed type of microstructure. Unaffected cladding zones show a dendritic microstructure. Except for one single coating dendritic microstructure shows significantly higher microhardness compared to heat treated microstructure. Values measured for that specimen produced with a CO$_2$/1%H$_2$ gas mixture does not fit in the general trend within the CO$_2$/H$_2$ series and is therefore regarded as outlier.

The standard deviation of microhardness values measured for one kind of microstructure is only 15 HV1 upon average. In contrast difference between the two types of microstructures typically exceeds 100 HV1. Only a relatively weak trend towards increased microhardness for addition of 25% oxygen to argon or carbon dioxide is observed, while higher oxygen contents in the gas mixture result in reduced microhardness. However, for mixtures of helium and oxygen such dependency cannot be confirmed. Carbon dioxide that has significantly lower oxidizing effect than oxygen only shows a relatively small hardness increasing influence for a content of 75% in mixtures with argon, helium or nitrogen. However, the use of pure carbon dioxide produces claddings with a dendritic microstructure that show the lowest microhardness among specimens produced with pure gases.

Only for use of the two-gas mixtures He/50%O$_2$ and CO$_2$/25%O$_2$ heat affected zones with equiaxed microstructure show a slightly increased microhardness, while the use of 75% oxygen content in mixtures with argon or carbon dioxide result in the lowest measured microhardness values.

Microhardness profiles reveal a clear correlation between weld penetration depth and sharpness of transition from cladding to base material hardness (Figure 7). Claddings with deep weld penetration that have been produced with e.g. pure argon or carbon dioxide rich mixtures show gradually decreasing hardness values for distances from cladding surfaces exceeding 1.5 mm. In contrast hardness profiles of claddings produced with oxygen addition to argon or helium show a drastic drop from about 700 HV0.1 down to about 250 HV0.1, which represents the base materials hardness, for distances from cladding surfaces exceeding 1.5 mm.

**Figure 6: Microhardness HV1.0 in areas with dendritic microstructure (red, no heat treatment effect by deposition of overlapping seams) and in areas that show transition towards an equiaxed type of microstructure (blue) depending on the applied process gas composition**

**Figure 7: Exemplary microhardness HV0.1 profiles of specimens produced with pure argon (green squares), Ar/50%CO$_2$ and (red diamonds), Ar/25%O$_2$ (blue circles) and He/50%O$_2$ (orange triangles) gas mixtures**

During production of specimens with a Ar/25%O$_2$ gas mixture and the Coax8 powder nozzle the melt pool emission is almost identical in comparison to the use of the PN6625 nozzle. In reproducibility tests with oxygen only contained in the laser process gas 219 and 222 units are measured, while use of a Ar/25%O$_2$ mixture both in the laser process and the carrier gas results in 221 units emission intensity. For use of the Coax8 nozzle 225 units are recorded.

Despite identical melt pool emission intensity, both the amount of deposited material and the weld penetration differ strongly for use of the two powder nozzles. In reproducibility tests with oxygen only contained in the laser process gas the weld penetration area in cross sections amounts to 4.3 and 4.8 mm$^2$ and for use of a Ar/25%O$_2$ gas mixture both in the laser process and the carrier gas 4.4 mm$^2$ is measured. In contrast the use of a Coax8 nozzle results in 9.5 mm$^2$ weld penetration cross section area. Also, the cross section area of deposited material amounts to only 17.2 mm$^2$, while the use of a PN6625 nozzle results in 20.6 and 20.9 mm$^2$ in reproducibility tests and
21.1 mm² for use of an Ar/25%O₂ gas mixture both in the laser process and the carrier gas.

Smaller cross section areas of deposited material is in good agreement with gravimetric powder usage data. While the use of a PN6625 nozzle results in 100% and 98.2% in reproducibility tests and 98.5% for use of a Ar/25%O₂ gas mixture both in the laser process and the carrier gas, only 84.5% powder usage are measured for use of a Coax8 nozzle. Decreased powder usage might be due to the bigger powder focus diameter and according loss of particles that are not injected into the formed melt pool. Such approach could also explain significantly stronger weld penetration as a consequence of reduced laser power transfer to powder particles and accordingly higher heat transfer to substrates.

Comparison of results for use of pure argon and a Ar/25%O₂ gas mixture shows different tendencies depending on the type of the applied powder nozzle. While adding oxygen to inert gases consistently causes a reduced weld penetration using a PN6625 nozzle, a significantly lower weld penetration, i.e. 6.9 mm² cross section area, is observed for the use of pure argon in case of a Coax8 nozzle. Also, a reduced area of deposited material, i.e. 16.0 mm², and a reduced powder usage, i.e. 81.3% are measured. Lower powder usage might be due to formation of a smaller melt pool and accordingly lower probability of particle injection into the formed melt pool.

Generally, microhardness of claddings produced with a Coax8 nozzle confirms the dependency of hardness on the extent of dissolution with base material. The use of pure argon results in comparable weld penetration areas, i.e. 6.7 mm² for a PN6625 and 6.9 mm² for a Coax8 nozzle. Also, microhardness in areas with dendritic microstructure and in heat affected zones is roughly the same. 604 HV0.1 / 478 HV0.1 and 625 HV0.1 / 503 HV0.1 are measured on specimens produced with a PN6625 and a Coax8 nozzle respectively. In contrast a weld penetration area of 9.5 mm² observed for use of a Coax8 nozzle with a Ar/25%O₂ gas mixture results in only 457 HV0.1 and 480 HV0.1 microhardness in the area of dendritic microstructure and in heat affected zones respectively.

Using a PN6625 nozzle and pure oxygen both as laser process and carrier gas result in a particularly small weld penetration area of only 4.1 mm², a relatively low powder usage of 92% and accordingly a small cross section area of deposited material, i.e. 19.1 mm². Despite a particularly low dissolution of base material microhardness in areas with dendritic microstructure and in heat affected zones amounts to only 515 HV0.1 and 511 HV0.1 respectively. That confirms once more a detrimental effect of excessive oxygen contents on powder usage and microstructure due to oxidation processes, but also a beneficial effect of oxygen on limiting dissolution with base material.

Conclusions

- Melt pool emission intensity increases strongly, when adding oxygen to argon, helium or carbon dioxide, while carbon dioxide addition to argon, helium or nitrogen only shows considerable effect for 75% carbon dioxide content.
- Use of high density gas mixtures, especially carbon dioxide rich mixtures, permits particularly high powder usage.
- Use of mixtures of the inert gases helium or argon with 25% oxygen results in improved powder usage, but for higher oxygen contents the powder usage decreases consistently. For CO₂/O₂ gas mixtures the powder usage drops with increasing oxygen content.
- In comparison to the use of pure argon, helium or carbon dioxide the addition of at least 25% oxygen results in decreased weld penetration depth for use of a PN6625 nozzle. In contrast the addition of 25% oxygen to argon causes the formation of a significantly deeper weld penetration for use of a Coax8 nozzle.
- The addition of 25% oxygen to argon or carbon dioxide as well as the addition of 50% oxygen to helium permit production of particularly hard claddings. For higher oxygen contents continuously decreasing hardness is observed. Besides an oxidation induced hardness loss also increasing dissolution with base material causes reduced cladding hardness.
- Zones that are subject to heat treatment by subsequently deposited seams show a transition from dendritic to equiaxed microstructure, which is accompanied by hardness loss of about 100 HV1.0.
- High reproducibility of laser cladding processes is proved at the example of a Ar/25%O₂ gas mixture.
- In tests with overall gas composition Ar/25%O₂ no difference between use of pure argon in carrier gas and increased oxygen content in the laser process gas or a homogeneous composition of carrier and laser process gas is observed.
- The use of CO₂/H₂ mixtures results in comparably strong porosity, which might be attributed to formation of water vapor.

Summary

Although laser cladding is carried out in conduction welding mode only the choice of process gases takes significant influence on the process characteristics. Besides the gases density that influences momentum transfer to powder particles and melt formed on substrates their thermal conductivity takes influence especially on cooling rate of formed melt pools. Additionally the chemical behavior of process gases can be exploited effectively. For cladding of nickel based self fluxing alloys especially oxygen permits limiting weld penetration and according alloying of claddings with base material despite oxidation reactions that cause a strong increase of melt pool emission intensity. If critical contents are not exceeded, there is a positive effect of oxygen on coating hardness. However,
this effect could not be confirmed when switching from a coaxial feed powder nozzle with six discrete powder injectors to an annular gap powder nozzle. Carbon dioxide tends to promote dissolution of base material in claddings. Mixtures of carbon dioxide and hydrogen promote pore formation.

The described findings cannot be transferred to laser cladding processes with different materials. Furthermore there are remaining questions e.g. concerning the influence of the powder nozzle’s design on the effect of oxygen addition. Therefore additional studies need to be conducted in order to achieve a better comprehensive understanding and finally to exploit the full potential of tailored process gases in laser cladding processes.

References


