3 Laser surface engineering

The subject of this chapter is the analysis of laser surface treatment processes that are applied to improve the surface properties of metals and alloys. First, the laser hardening technique is evaluated in terms of the microstructure transformations in gray cast iron, a material that is widely used in automotive engines. Then, the laser cladding processes with the coaxial and the side setups are evaluated by empirical and statistical analyses to determine the optimal process window that prioritizes the clad quality and the efficiency of the technique.

3.1 Laser surface engineering techniques

Laser surface treatments have been investigated extensively. The oldest paper in laser surface remelting of cast iron dated from 1969 and concentrated on the track geometry and surface topography [1]. Cracks, pores and other defects at the melted surface were always a problem and they still are to date. Nowadays laser surface hardening is used for many applications to improve the mechanical properties. Considerable research was done on the mechanical properties of the modified layers. The most common property to be measured is the micro hardness and this was described in nearly all the papers on laser surface treatment. Other properties like the wear- and corrosion resistance have also been thoroughly investigated. It is known that the wear resistance improves with a factor of 2 after a pin-on-disc type wear test and others investigated the roughness at various stages of running-in wear on the laser treated material [2,3].

When a high power laser is used as a heat source, the surface of materials can be improved by processes that are grouped in two categories. The first is a method when no additional material is deposited on the surface of an alloy and the improvement of properties is achieved via phase transformations. That is the case of the laser hardening and the laser remelting techniques. The other category is where an additional amount of a more resistant material is added to the surface of a less resistant alloy by the so called laser-melt particle injection and laser cladding technique that exists in the coaxial and the side way injection of particles. There are many advantages in the utilization of these techniques as for example: the treatment of hard-to-reach surfaces, narrow or strange shaped surfaces, accurate control and
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reproducibility. For the success of each technique, it is essential to choose the right combination of parameters. However, due to the complex interactions of the laser with different materials and the diverse application mode to find the optimal application conditions is not always a straightforward task. This work aims at showing how the application of three different modes of laser surface engineering techniques (laser hardening of gray cast iron, laser cladding with the coaxial and the side setup) can be analyzed for its improvement.

3.1.1 Laser transformation hardening and remelting of grey cast iron

The improvement of the materials properties can be done by transforming its microstructure by a controlled application of heat. Independently of the technique used for the application of heat, the most important factor is material related, that is, the alloy system that is being investigated and the possible phase transformations. It is necessary, therefore, to study and to describe the alloy system before the material is processed.

In this work, gray cast iron was investigated. Gray cast iron is widely used in engines because of the low price, good castability and excellent machinery desirable in the finishing of parts. Pressure by the European norms to reduce the pollution and the demand for high performance engines provides an impetus for the improvement of the mechanical properties. Figure 3.1 is the phase diagram of the Fe-Fe₃C in equilibrium [4] system that provides information about all possible phase transformations in the steel and cast iron compositional ranges under thermodynamic equilibrium conditions. Based on the data of the diagrams, the process can be tailored according the transformation temperatures so that the desirable phases are formed.

![Figure 3.1: Binary Fe-Fe₃C Phase diagram and the unit cells structures of austenite and ferrite.](image-url)
Figure 3.2 shows what is expected when an ideal laser transformation hardening process is applied to steel. While a high power laser scans the surface of the material its surface heats up. The surface temperature is then measured and controlled by a device (as for example, a pyrometer) so that the desirable phase transformations take place. Assume that the initial microstructure is composed of ferrite and pearlite (a fine lamellar structure of alternating platelets of ferrite and iron carbide). During slow heating the ferrite and the pearlite will be transformed to austenite above 726 °C. As the laser beam moves away, the initially heated region cools down rapidly due to heat conduction into the metal and martensite is formed increasing the hardness at and near the surface of the piece.

Another process that also leads to the improvement of surface properties is performed when the transformation temperature is attained and the melting temperature is reached, for steel that is around 1150 °C. In this case, a part of the material adjacent to the surface melts and resolidifies when the laser beam has moved away. Due to the melting and resolidification process, usually the crystal growth is quenched due to rapid cooling and therefore a refined structure with improved hardness and wear resistance is attained. This surface treatment is the so-called laser remelting.

Cast irons embrace a large family of ferrous alloys. Cast iron alloys contain more than 2 wt % carbon and 1 to 3 wt % silicon. Wide variations in properties can be achieved by varying the balance between carbon and silicon, by alloying with metallic and non-metallic elements, and by varying melting, casting and heat treating practice. Cast irons, as the name implies, are intended to be cast for shaping the product rather than formed in the solid state. The advantages of cast irons are low melting temperatures. They are very fluid when molten, do not form undesired surface films when poured and they undergo slight shrinkage during solidification and cooling. However, cast irons have relatively low impact resistance and ductility, which may limit their use.

Mechanical properties, like strength and elasticity of cast irons depend strongly on structure and distribution of microstructural constituents. The damping capacity, thermal conductivity and other physical properties are mostly influenced by the microstructural feature called free graphite. Shape and distribution of free graphite are more important than composition for cast irons. In many instances, iron of a given composition can be made into four basic types by varying casting or heat treating practice. The four basic types of cast irons
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are white iron, gray iron, ductile white iron and malleable iron. White and gray cast iron derive their name from the appearance of their fracture surface. White cast iron is formed when carbon in the molten iron does not form graphite but remains combined with iron, often in the form of massive carbides. In the fracture surface these carbides are seen as white regions giving white iron its name. Gray cast iron on the other hand reveals a gray fracture surface. The reason for the gray fracture surface is that carbon in the material form large graphite flakes and fracture paths follow these flakes. The fracture surfaces appear gray due to the predominance of graphite flakes. Ductile iron derives its name by the fact that in cast form it has a measurable ductility. White and gray cast iron are not very ductile in tensile tests. Malleable iron is cast as white iron which is heat treated to improve the ductility of the otherwise brittle material. The material in our experiments is of the type gray cast iron, with the commercial name GG25 [5]. Its composition is given in Table 3.1.

<table>
<thead>
<tr>
<th>Element</th>
<th>wt %</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>3.30 – 3.55</td>
</tr>
<tr>
<td>Si</td>
<td>1.80 – 2.20</td>
</tr>
<tr>
<td>Mn</td>
<td>0.50 – 0.90</td>
</tr>
<tr>
<td>P</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>S</td>
<td>&lt; 0.13</td>
</tr>
<tr>
<td>Cr</td>
<td>0.20 – 0.40</td>
</tr>
<tr>
<td>Cu or Ni</td>
<td>0.40 – 0.60</td>
</tr>
</tbody>
</table>

Except for carbon, Si and P determine the metallurgy of cast iron affecting the condition and the kinetics of carbide formation on cooling. The carbon equivalence (CE) is a method that often is used to simplify evaluation of the effect of composition in cast irons and it is defined as:

$$ CE = C + \frac{1}{3} (Si + P) $$

Comparison of CE with the eutectic composition in the binary Fe-C phase diagram will indicate whether a cast iron will behave as a hypoeutectic or hypereutectic alloy during solidification. When the CE is near the eutectic value, the liquid state still exist at a relatively low temperature and solidification takes place over a small temperature range. This is important for the uniformity of the properties when casting. According to the binary phase diagram is the eutectic point at a CE of 4.30 wt %. This would mean that the solidification process of GG25 behaves as an eutectic material.

Seven types of graphite precipitates are established by the American Society for Testing and Materials (ASTM) [5]. The graphite in our cast iron is of type VII, flake graphite. Gray cast iron has several unique properties that are derived from the existence of these graphite flakes in the microstructure. It can be machined easily and it has outstanding properties for applications involving vibrational damping and thermal shock. Flake graphite is formed with
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low cooling rate with enough time for carbon to diffuse and form flakes that can be subdivided into 5 types which designated by the letters A through E. Type A is the most uniformly distributed, randomly oriented and it is superior to other types of flakes in certain wear applications and internal combustion engines. It is good for vibrational damping, impact resistance and thermal conductivity. GG25 cast iron has type A graphite flakes in a pearlite matrix. According to the stable binary Fe-Fe$_3$C phase diagram associated with extremely slow cooling, the iron and carbon would solidify to graphite flakes in a ferrite matrix. The matrix of GG25 cast iron is pearlite with lamellae of ferrite and Fe$_3$C (cementite). An explanation of a pearlite matrix can be found in the metastable binary Fe-Fe$_3$C phase diagram. This phase diagram is associated with a reasonable cooling rate with time scale that not all the carbon can diffuse. The mechanisms of solidification are now discussed.

As the temperature decreases, so does the carbon solubility in the austenite. The carbon atoms are diffused outwards from the austenite to graphite flakes and a new Fe$_3$C phase. The carbon solubility decreases even dramatically when austenite is transformed to ferrite. Ferrite can only contain a very small amount of carbon and therefore the carbon atoms are diffused to the tips of the adjacent cementite lamellae. Similarly are the Fe atoms rejected from the cementite to the ferrite. As the temperature decreases, the matrix will undergo an eutectoid transformation. Figure 3.3 shows how ferrite and cementite can grow cooperatively during the transformation front.

![Figure 3.3: Growth of eutectoid front with diffusion in austenite.](image)

This mechanism is concluded from the experimental observation by the fact that no isolated cementite is observed but only lamellae cementite. The complete dissolution of the metastable cementite is prevented by a fast cooling rate. Because a lot of free carbon in the form of graphite flakes or globules are present, the laser cladding on cast iron is not easy and therefore also not very frequently reported in the literature [6,7,8,9].

The process of hardening of cast irons involves the formation of the metastable martensite phase which is extremely hard and brittle if compared to ferritic and austenitic phases. The transformations at heating and cooling leading to the formation of martensite are described with the aid of the binary Fe-Fe$_3$C phase diagram, Figure 3.1.

First, the transformation from ferrite to austenite upon heating will be described. The pearlite consists of lamellae of ferrite and cementite. As the pearlite is heated up, a transformation of ferrite to austenite occurs at 912 °C, that is, the crystal structure changes from body centered cubic (bcc) to face centered cubic (fccc). If the heating rate is high, the
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ferrite would directly transform to austenite without diffusion of large amounts of carbon from cementite. The next step involves the transformation at cooling. At low cooling rate austenite would transform back to ferrite. A remarkable phenomenon happens at high cooling rate. In such a situation the atoms are trapped in a metastable phase as a result of the short time lapse to rearrange to the original configuration. It may be thought as a transformation product that is competitive with pearlite and the bainite. The martensitic transformation occurs when the cooling rate is high enough to prevent carbon diffusion.

The martensite phase transformation can be illustrated at the atomic level. It is known that austenite (fcc) experiences a polymorphic diffusionless transformation to body centered tetragonal martensite (bct) under the growth speed of approximately 1000m/s [10]. E. C. Bain demonstrated in 1924 his model for the fcc→bct model, Figure 3.4.

Figure 3.4: Overview resembles A) fcc, B) bcc and C) bct lattices in the Bain transformation [11].

In three steps of Figure 3.4 it is shown how the bct lattice can be obtained from the fcc structure with a minimum of atomic movements and a minimum of strain in the parent lattice. In B it is shown how an elongated unit cell of bcc can be drawn within two fcc cells. In C the transformation from bct to bcc unit cell is achieved by a contraction of 20% of the length c and an elongation of 12% in the length a. All the carbon atoms remain as interstitial impurities in martensite, the possible positions of the carbon atoms are marked by x in the bct unit cell.

The cooling rate is such that the majority of carbon atoms in solution on the fcc γ-Fe remain in solution in the α-Fe, making the martensite actually a supersaturated solid solution of carbon in ferrite. This super saturated solid solution is in theory capable to transform to another structure when the diffusion rate becomes sufficient. At room temperature the martensite is almost fully stable. However, as mentioned, martensite and pearlite grow in competition when the austenite is cooled down; therefore in order to create martensite, the formation of pearlite has to be prevented under a certain cooling rate. To visualize the influence of time and temperature on the microstructure a Time Temperature Transformation reaction diagram (TTT diagram) can be used. Figure 3.5 is a TTT diagram for a cast iron with chemical equivalent to the composition of the cast iron, GG25, used in this work [12]. To prevent the formation of pearlite, the cooling rate has to be such that the pearlite transformation can not start. This minimum cooling rate needed to form martensite is derived as a straight line in the TTT diagram, which is approximately 70 °C /s.
Figure 3.5: TTT diagram of cast iron with a chemical composition similar to GG25 predicts the martensitic transformation above 70°C/s [12].

The main advantage that justifies the application of the laser surface hardening is that the cooling rates are extremely fast due to the high temperature gradient applied locally on a small volume at the surface of a relatively massive and cold workpiece. Furthermore, the cooling rate is simply controlled by the scanning speed that moves the heat source away. As a result the hardness achieved by this process is usually very high. The challenge is to control the surface temperature during hardening. The surface temperature can be measured by a pyrometer and a feedback system controls the laser power to keep the surface temperature on the required level. In this way, a control of temperature can result in a more homogeneous hardened zone.

3.1.2 Laser cladding

Laser cladding is a technique that allows the deposition of thick protective coatings on weaker substrates. The process can be described as the addition of one material by alloying on the surface of the substrate, where the heat source is a high power laser beam. The resulting thickness of the clad corresponds to the molten region volume, which is typically 50 µm to 2 mm in one step. Once a thicker protection layer is needed, the process can be applied again.

The laser cladding process can be executed in three different ways. Using the pre-placed powder the alloying material is applied in the form of slurry [13,14]. This process has usually a small processing window, is time-consuming and also sometimes difficult to adapt on complex geometrical shapes. Secondly, the clad material can be delivered by wire feeding to the melt pool. This process is difficult to control, which usually leads to high dilution rates. Finally, the laser cladding by powder injection is an attractive methodology and is the subject of this section.
In this process a carrier gas is used to form an alloying powder stream blown under the laser beam while it scans the surface of the substrate generating a melt pool with a depth that corresponds to the thickness of a single clad generated in a single step. A fully dense layer can be achieved when single tracks consecutively overlap side by side. The process requires minimum surface preparation and solves the problem of application in complex geometries. There are two ways of feeding powder: from the side or coaxially to the laser beam, see Figure 3.6.

![Figure 3.6: Coaxial (left) and side (right) laser cladding set-ups above a moving substrate.](image)

The technique of side-cladding has been extensively described in previous works [15,16]. When the powder stream is injected off-axis from the laser beam, the change of substrate movement direction leads to completely different local cladding conditions. For instance, a so-called “against hill” cladding condition takes place when the powder stream is applied from the side from which the substrate moves. In this case, the clad powder is trapped temporarily in a corner formed between the molten track and the flat substrate. This leads to higher powder efficiency than in the so called “over hill” cladding set-up, when the powder is fed from opposite side [13,17].

When the powder stream is delivered coaxially with the laser beam, all directions of the substrate movement in a plane perpendicular to the laser beam are equivalent. The coaxial laser cladding process is thus independent of the cladding direction. Therefore, it is possible to produce equivalent tracks independently of the moving direction of the work piece. The advantage of coaxial laser cladding is employed in the formation of metallic parts from 3D designs [18,19].

### 3.1.3 Characterization of Nd-YAG laser energy distribution

In all the experimental works presented in this thesis a Nd-YAG laser (2kW, Rofin Sinar CW 20) was used, Figure 3.7. The great advantage of this laser is that the beam can be transported through optical fibers because of the small wavelength of 1.06 µm. The active medium is solid Neodymium-Yttrium Aluminum Garnet. Although the laser operates in a pulse mode, a near continuous mode can be obtained by using a high frequency (100Hz). After the beam leaves the fiber it is going through a 200 mm lens system. The spotsize of the
beam can be adjusted by changing the out-of-focus distance. Argon gas is used as a shielding gas to prevent oxidation of the material and to protect the lens. The characterization of energy distribution as a function of the out of focus distance is done with PRIMES Focus monitor. In this device a ceramic needle moving with a high speed (1875 rpm) through the beam and it measures the intensity. The results are given in Figure 3.8 showing the normalized beam energy distribution as a function of the focus distance.

The beam has a top-hat distribution near the focus point and gets a Gaussian distribution far from the focus point. This Gaussian distribution is taken into consideration in further analysis for the modeling of the laser beam. The radius of the beam is defined as the value where the intensity is 1/e of its maximum value in the centre. Measurements have shown that the formula for the calculation of the beam radius is

\[ r_b = 0.09D, \]

where \( r_b \) is the beam radius and \( D \) is the defocus distance.
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3.2 Laser hardening of grey cast iron

3.2.1 Experimental

In laser surface hardening experiments the temperature range between 911.5 °C ($\alpha \rightarrow \gamma$ transformation) and 1150 °C (melting point) should be kept. Laser hardening experiments are performed with the temperature monitored by a pyrometer Maurer type KTR 1075 with a linear conversion of signal voltage of 0 to 10 V corresponding to a temperature range of 400 to 1500 °C. A computer interface allows the control of the temperature during laser surface hardening by responding to the pyrometer measurement with the variation of the laser power $P$, via a linear change of laser lamp pumping power inside an interval of 200-1750 W, Figure 3.9. Two different laser defocus distances are used which resulted in two different laser beam diameters. The defocus distance of 30 mm results in a laser spot with radius $r_b = 2.7$ mm and a defocus of 15 mm results in a radius of 1.35 mm. The laser power was gradually increased during the formation of single tracks at scanning speeds of 0.5, 2, 5, 10, 15, 25 and 50 mm/s with the two laser beam diameters, totaling into 12 single track samples.

![Figure 3.9: A typical temperature and laser power vs. time graphs in the transformation range. The straight line is the laser power. $T_1$, $T_2$ and $T_3$ are 1000, 1075 and 1150 °C respectively.](image)

With a radius of 2.7 mm the required temperature range for hardening was only reached at scanning speeds from 0.5 to 15 mm/s. The lowest scan velocity was not taken into account because the pyrometer measurements presented too many fluctuations, while under highest scan speed the desired temperature range could not be reached. For the radius of 1.35 mm, scanning speeds of 5 to 50 mm/s resulted in the laser tracks within the desired temperature range.
3.2.2 Geometry of hardened tracks

The influence of the surface temperature and the scan speed on the hardened area were investigated. It was chosen to examine the cross sections of the material that experienced temperatures $T_1= 1000^\circ C$, $T_2= 1075^\circ C$ and at the end of the transformation temperature $T_3= 1150^\circ C$. An SEM micrograph of a laser track cross section is given in Figure 3.10 with an indication of the depth and width that were measured. The positions of the interesting temperatures are marked on the single tracks and cross sections are made. Eight different laser tracks with three cross sections each are selected, therefore 24 cross sections are necessary to explore the influence of the scan speed and surface temperature on the track geometry.

![Figure 3.10: SEM micrograph of laser hardened track with indication of depth and width.](image)

Figure 3.11 brings the results of the laser track depth vs. temperature given for laser beam radius $r_b= 2.7$ mm and $r_b= 1.35$ mm respectively.

![Figure 3.11: A) track depth vs. surface temperature graphs, $r_b= 2.7$ mm. B) track depth vs. surface temperature graphs, $r_b= 1.35$ mm](image)

From the plots the track depth increases almost linearly with the temperature for the all scan speeds at both laser beam radius. The tracks vary in depths from 250 µm at the highest scan
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Speed to depths of 500 µm at the lowest scan speed for hardening with beam radius \( r_b = 1.35 \text{ mm} \). Hardening with \( r_b = 2.7 \text{ mm} \) results in tracks with depths that vary from 500 µm, at highest speed, to 800 µm, at lower scan speed. The slopes of the fitted lines are nearly equivalent to each other, except the slope for the scan speed 2 mm/s. At this speed, the laser power applied was around 400 W, which is low and therefore difficult to keep at a stable level. The slopes of the fitted depth vs. temperature curves of hardening are nearly equivalent. This would mean that the increase in track depth per increase in surface temperature is equivalent in all scanning speeds. The interaction time is simply the beam diameter divided by the scan speed. The graphs show that the track depth strongly depends on the interaction time: an increase in interaction increases the track depth.

*Figure 3.12* shows the width of the track and the corresponding surface temperature. From the plots a linear relationship is observed between the track width and the surface temperature. Hardening with a beam radius of \( r_b = 2.7 \text{ mm} \) results in variations of width from 3.5 mm at low surface temperature to 4.8 mm at high surface temperature and high scan speeds. The line width of the scan speed 2 mm/s has a lower slope than those of speeds 10 and 15 mm/s. In the case of hardening with a beam radius 1.35 mm the variation of track width is from 2 mm at low surface temperature to 3.1 mm at high surface temperature and high scan speeds. Another observation of the graphs is the fact that the track width is nearly constant for all scan speeds at surface temperature of 1000 °C. For the radius 2.7 mm, the track width at 1000 °C is around 3.6 mm and for the radius 1.35 mm, approximately 2.1 mm. The steepness of the curves increases as the scan speed increases. At high scan speeds also the power is high so as to maintain the surface temperature.

![Figure 3.12: A) track width vs. surface temperature graphs, \( r_b = 2.7 \text{ mm} \). B) track depth vs. surface temperature graphs, \( r_b = 1.35 \text{ mm} \)](image)

3.2.3 Characterization of hardened gray cast iron

Microstructure of untransformed zone.
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From literature [5] it is known that GG25 cast iron has a microstructure with pearlite matrix and randomly oriented and distributed graphite flakes. The Figure 3.13 brings SEM and optical micrographs of the GG25 gray cast iron.

Figure 3.13: A) SEM micrograph of un-etched cast iron showing the graphite flakes, B) Optical micrographs of nital etched cast iron reveal pearlitic microstructure and MnS particles, indicated by arrow.

In order to check the degree of success of the laser surface hardening the microstructure is characterized by XRD analysis and SEM-EDS. X-ray diffraction (XRD) with $\lambda_{\text{CuK}\alpha} = 1.542$ Å energy in the range of 20: 20-120° was performed on the base material and on the surface of the hardened track, Figure 3.14. The phases are identified according to the Powder Diffraction Database (PDF) and to an article of Zarubova [20].

Figure 3.14: XRD spectra of transformed material surface and base material.
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The XRD spectrum, associated with the EDS and SEM analyses, of the non-treated material indicates the presence of the following microstructural features in the substrate: ferrite, cementite, MnS, steadite and graphite. If this pattern is compared with pattern of the hardened material it can be seen that some retained austenite was formed. The material melts are due to high carbon content > 1%, the martensite starting temperature decreases below room temperature and austenite is retained. Martensite peaks are compared with theoretically calculated peaks of bct crystal diffraction with lattice parameters involving carbon content of 1.8 wt%. The positions of the peaks are similar. A strong peak at $2\theta = 31.6^\circ$ is identified as iron oxides, $\text{Fe}_2\text{O}_3$.

Microstructure of transformed zone.

Microscopic observations show that the hardened tracks are composed of three regions, Figure 3.15. There is an indication melting present at the track surface, even in samples with a measured surface temperature of 1000 °C. Melting is not desired in this case because of the residual stress that may cause cracks and roughening of the surface, which requires further polishing and grinding before use. Melting was always in the middle of the track surface. This can be explained by the fact that due to the circular beam shape the time spent under the centre of the beam is longer than at the sides. The Gaussian energy distribution provokes a higher temperature in the centre of the beam. The pyrometer measures the temperature over a certain area and provides only an average temperature. The real surface temperature in the track centre is higher than the temperature indicated by the pyrometer.

![Figure 3.15: Optical micrograph of laser hardened track with three zones. Scanning speed 5 mm/s, $r_b = 1.35$ mm and $T_{surf} = 1075$ °C.](image)

The transformed region is the area where the graphite flakes are left intact and where the pearlite matrix has transformed. This region clearly differs in microstructure from the non-transformed base material. A remarkable structural observation is the fine stripping, representing the cementite lamellae. This is observed throughout the whole transformed zone sometimes up to the melt boundary. The boundary between the base material and the transformed region is quite sharp and irregular due to the lamellae of ferrite and cementite.

The microstructure between the transformed and non-transformed boundary consists of residual cementite which remains undissolved during the heating period, revealing that the
heating rate was high. Between the residual cementite fine martensite plates are observed, see Figure 3.16. Relevant information about the transformation mechanism during heating can be taken from these micrographs. It shows evidence of transition from ferrite to martensitic plates located between the cementite lamellae.

![Figure 3.16: Sample processed at 30mm/s. A) Boundary between the transformed and untransformed zones. B) Martensite plates between Fe₃C (cementite) plates.](image)

Due to the increasing peak temperature and a longer heating time, the content of dissolved carbon in austenite increases. As a consequence the martensite morphology and the amount of retained austenite changes significantly throughout the hardened track. From the boundary with the untransformed zone towards the surface the cementite plates tend to decrease its thickness until it is completely dissolved. Still in the non-molten zone it is possible to observe traces of the original pearlitic structure. This apparent pearlite is known as ghost pearlite in retained austenite, Figure 3.17. Although carbon has diffused from cementite, the original pearlitic structure indicates that chemical heterogeneity is present. The presence of one phase is proven by the fact that the martensite plates pass through several lamellae in the ghost pearlitic matrix in, for example, a sample made with laser scan speed of 30 mm/s.

![Figure 3.17: Martensite plates are able to cross the ghost of pearlite in the transformed zone.](image)
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Microstructure of resolidified zone.

Towards the melted zone, martensite plates become continuously different. An optical micrograph of the resolidified molten zone show large martensite plates with the characteristic needle structure, Figure 3.18. In this region of the transformed zone the martensite plate become larger and more pronounced due to the large amount of diffused carbon into the austenite. The temperature increases the closest one gets to the surface. Once the cementite plates are dissolved the growth of the martensitic plates is not hampered any longer. Midrib martensite needles form typical chains observed in the molten area. This structure is characteristic for twin martensite with high carbon content [20]. Furthermore the density of the martensite plates increases close to the graphite flakes since these are good thermal conductors. On the top and centre of the molten surface the microstructure consists mainly of retained austenite. The graphite flakes are dissolved as a result of the laser interaction.

Figure 3.18: Melt with retained austenite dendrites and martensite midribs (on the top), on the transformed zone graphite flakes are present and there is a high concentration of martensite plates.

3.2.4 Transformation hardening mechanism

The kinetics and mechanisms of phase transformation during heating of pearlite to austenite can be described with a binary Fe-Fe₃C phase diagram and a continuous heating transformation (C.H.T.) diagram in the following figure.
Figure 3.19: C.H.T. diagram representing the kinetics of pearlite to austenite transformation at low \((U<U'_{L})\) and high \((U>U'_{L})\) heating rates. \(A1\) and \(A2\) given in Fig. 3.1.

The C.H.T. diagram describes the influence of heating rate on transformation mechanism. The lines \(U_{L}\) and \(U'_{L}\) represent high and low heating rates respectively. \(\gamma^*\) is the area of inhomogeneous austenite. It is known that the pearlite \(\rightarrow\) austenite transformation in laser surface hardening can occur by two competing mechanisms:

1) by heterogeneous nucleation and growth depending on the diffusion of carbon. The \(\text{Fe}_\alpha(C) + \text{Fe}_3\text{C} \rightarrow \text{Fe}_\gamma\) transformation occurs and carbon plays a certain role in this transformation. The transformation takes place in a temperature interval between \(A_1=723^\circ\text{C}\) and \(A_3=912^\circ\text{C}\) at low parts \((U<U'_{L})\) of the C.H.T. diagram representing low heating rates and temperature gradients. Nucleation of austenite takes place if the time of pearlite between \(A_1\) and \(A_3\) temperatures exceeds the incubation time for nucleation. This mechanism depends on the dissolution rate of cementite and diffusion rate of carbon in austenite. This mechanism is present at slow heat treatment.

2) by propagation of the \(\alpha/\gamma\) interface. The direct \(\text{Fe}_\alpha ( + \text{Fe}_3\text{C}) \rightarrow \text{Fe}_\gamma ( + \text{Fe}_3\text{C})\) transformation occurs and carbon and cementite play a minor role in this transformation. The beginning of this transformation takes place at the upper part of the C.H.T. diagram and therefore the transformation shifts to the temperatures higher than \(A_3\) temperature, see temperature \(T_1\) in Figure 3.20. This transformation happens at high heating rate and depends on the diffusionless transition of ferrite to austenite.

Which of the two mechanisms dominate depends on the temperature gradient and the scan speed or in other words the heating rate. Usually in laser transformation hardening there is a combination of the two mechanisms.

Microscopic observations on laser treated samples reveal the presence of undissolved cementite near the boundary with the untransformed phase, Figure 3.16. This means that
transformation mechanism 2) dominates the transformation with a shift of the transformation temperature above $A_3$ temperature. Observations show that even at the lowest scan speed of 2 mm/s the direct $\alpha \rightarrow \gamma$ transformation was dominant over the diffusion controlled mechanism. In Figure 3.20 is an illustration of the dissolution of cementite after the $\alpha \rightarrow \gamma$ transformation according to the heating line $U=U_L$ and the various stages of temperatures.

![Figure 3.20: Stages in temperature of cementite dissolution at high heating rates $U=U_L$.](image)

Phase transformation $\alpha \rightarrow \gamma$ starts at $T_1$ and finishes at $T_2$. This microstructure is desired because of the high hardness, small stresses and isotropic properties due to the random orientation of the martensite needles. At this point the undissolved cementite and austenite coexist. As the temperature rises, carbon will diffuse from the cementite to the adjacent austenite giving rise to a carbon concentration gradient in $T_3$. Until $T_4$ there is the phenomenon of phase heterogeneity. Cementite disappears after $T_4$ but there is still a carbon gradient showing the stripping, chemical heterogeneity at $T_5$. As the temperature increases, there will be a homogenization in austenite.

If austenite is cooled fast enough to prevent the formation of pearlite then a certain undercooling can be reached at which the fcc structure loses its mechanical stability and forms a distorted bcc lattice (bct) by shear. The temperature at which this diffusionless martensite transformation starts is known as the $M_s$-temperature. It is known that the $M_s$-temperature decreases with increasing carbon content [21] and can become below room temperature. That is the reason for the presence of a fully austenite region near the melt with martensite needles.

3.2.5 Hardness characterization of laser hardened grey cast iron

The hardness is the most important physical property in laser surface hardening. To prove the effect of the process Vicker hardness measurements were performed. According to the literature gray cast iron has a hardness of approximately 250 HV. The microhardness was
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measured on perpendicular cross-sections of laser tracks in two directions: track depth and width. To have a representative value of the micro hardness, 3 arrays of 13 indents in the depth are measured. Measurements in the width are done at 2 arrays of 25 indents. Indents influenced by graphite flakes were omitted.

The measurements are done with a maximum load of 2N for 15 seconds. Samples treated with a laser beam radius \( r_b = 2.7 \, \text{mm} \) with surface temperature of \( T_{\text{surf}} = 1000 \, ^{\circ}\text{C} \) at scan speeds of 2, 10 and 15 mm/s are taken to measure the microhardness. Figure 3.21 is an optical micrograph with micro indents at the matrix and graphite flakes in the transformed zone.

![Figure 3.21: Optical micrograph with micro indents on the transformed zone.](image)

From the hardness plots, Figure 3.22, it is observed that the average hardness increased from 250 to about 600-900 HV. The variation of hardness depends on the location that the Vickers indenter touches the sample. When a higher content of martensite is probed, a higher value of hardness is registered.

![Figure 3.22: Hardness profiles in samples processed under different processing speeds.](image)
Chapter 3

3.3 Analysis of coaxial cladding process

Laser cladding is governed by complex interactions between laser beam, powder and substrate. Achieving a high precision in the control of the main process parameters is possible but requires a full understanding of the clad formation. The laser cladding process has several particular characteristics that govern the process, as for example, shielding gas flow rate, energy profile of the laser beam, pulsed or continuous laser beam. However, some of these are difficult to measure or to control. In brief, effects of the principal four process parameters are the following. Laser power $P$ quantifies the intensity of heat. In combination with substrate scanning speed $S$ the parameter $P/S$ is formed, which is related to the heat input. The powder-feeding rate $F$ controls the amount of material delivered, i.e. the quantity $F/S$ corresponds to the amount of powder delivered per unit of clad length. When a powder cloud is present under the laser beam, the heat transfer on the substrate is attenuated because of the shielding effect due to the powder [18]. Due to the path and time spent under the laser beam, coaxial feeding promotes the maximum laser beam attenuation, which may cause a shift of the minimum laser power necessary for melting the substrate or substantially change of the laser beam energy distribution inside the laser spot. The velocity of the particles $v_p$ is mainly controlled by the flow of the carrier gas. Usually argon is used as carrier gas as well as shielding gas to protect the clad from oxidation. However, $v_p$ is not so easy to measure [22]. Therefore the carrier gas speed at the nozzle opening is often used as an upper estimate of the particles speed.

Single laser tracks can be deposited for quite a very broad operational range of the processing window. A complete clad layer is formed by a successive deposition of single tracks side by side. However, if one wants to build up an optimal clad layer (well-bonded, thick, dense and without cracked) the single tracks should fulfill certain geometrical features, which can be achieved within a particular processing window.

In this part a theoretical and experimental study of the laser cladding process coaxial and side set-ups are presented aiming at understanding the basic concepts of the process and to understand the relations between the main laser cladding parameters and geometrical characteristics of an individual laser track.

The operational window for laser cladding process is usually defined in terms of laser power $P$ [W], laser beam scanning speed $S$ [mm/s] and powder feeding rate $F$ [mg/s]. Certainly, several others process parameters play a role, such as: laser beam spot size, laser beam energy distribution, sort and amount of shielding and carrier gas, size, speed and feeding direction of powder particles, etc. A complete description of the laser cladding process is rather complex, because of numerous of interactions (laser beam/powder stream, laser beam/substrate surface, powder stream/melt pool, powder stream/solid substrate, etc.) and physical phenomena are present. Therefore, the experimental examination of the process parameters window and a search for relations between the clad track characteristics and processing parameters are still necessary for exploration of new coating/substrate combinations.
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It is necessary, therefore, to explore the laser processing window in terms of $P$, $S$ and $F$ parameters and relate them to the main single-track geometric features reducing the needs for extensive and time consuming experiments. Laser cladding experiments with gradually increased laser power were performed to deposit superalloy powders on ferrous substrates. In this procedure single clad tracks are produced using constant beam radius, processing speed and powder feeding rate along the whole laser track length. The clad quality is characterized quantitatively by several geometrical parameters measured on the cross-section of these tracks. The relations between the processing parameters and the geometrical parameters of the transverse cross section are evaluated using statistical analysis and laser cladding processing map based on this relations was constructed.

Prior to the experimental part, however, an attempt is made to describe the melting regime of the particle in the coaxial laser from a theoretical viewpoint.

3.3.1 Theoretical calculation of particle melt regime in the coaxial laser setup

Figure 3.23 shows the relevant geometric features of the coaxial laser process. The distance between the coaxial nozzle output and the cladding surface $N_d$ is expected to be approximately 13 mm, due to the fixed shape of the powder cone escaping from the nozzle outlet (diameter of 9.5 mm under an angle of $\theta=70$ degrees). The focal point of the laser beam is for the coaxial set-up usually positioned close to the nozzle output so as to secure the nozzle by minimizing the laser beam radius near it. The laser beam diverges from the focus point in angle $2\varphi = 13.7$ degrees. These two angles, together with the laser defocus distance, $L_d$, and the nozzle working distance, $N_d$, are the input variables required to calculate the distance, $p_d$, over which powder particles are interacting with the laser beam.

Figure 3.23: Coaxial laser cladding geometry used in experiment and corresponding scheme for the calculation of the powder-laser beam interaction area.

The laser power required to melt the substrate and powder, $P_{ms}$ and $P_{mps}$, respectively, were calculated on the basis of a model suggested by Jouvard et al [23]. The basic assumptions are:

- the incident energy on the substrate contains two contributions: the energy coming from the transmitted beam and the energy coming from particles heated by the laser beam
inside the interaction area. Only a fraction of particles, which contributes to the clad layer, is assumed to transmit the thermal energy to the substrate;

- the laser beam is attenuated inside an interaction area by the particle cloud according to Lambert-Beer’s law \[24\]. A coordinate, \(z\), is measured from the point where the interaction between the particle cloud and the laser beam starts (see Figure 3.23);
- the spherical powder is homogeneously dispersed throughout the cross-section of interaction area but its concentration decreases with \(z\) because of increasing radius of the interaction area;
- the particle velocity inside the interaction zone is constant;
- heat transfer at the surface of the substrate follows a one-dimensional, semi-infinite wall approximation.

Under these constraints the laser power required to heat the substrate to the melting temperature \(P_m\) can be written as:

\[
P_{ms} = \frac{\sqrt{\pi} k_s (T_{ms} - T_{i_s})}{2\beta \sqrt{\alpha_s t_{int}}} \tag{3.3}
\]

and the laser power required to heat the powder to the melting temperature is:

\[
P_{mp} = \frac{m_p C_p (T_{mp} - T_{ip})}{\gamma} \tag{3.4}
\]

using:

\[
\beta = \frac{A_s}{S_{int}} \exp\left(\frac{-\varepsilon F p_d}{\pi \nu_p (r_n^2 + r_n p_d \tan \phi)}\right) + \frac{3 A_{pe} F}{4 S_p S_{int} \nu_p \rho_p} \gamma \tag{3.5}
\]

and

\[
\gamma = \frac{S_p A_r}{\pi \nu_p} \int_0^{r_p} \frac{1}{r_n^2 + rz \tan \phi} \exp\left(\frac{-\varepsilon F z}{\pi \nu_p (r_n^2 + rz \tan \phi)}\right) dz \tag{3.6}
\]

where: \(T_{ms}\) is the substrate melting temperature, \(T_{i_s}\) is the substrate initial temperature, \(k_s\) is the substrate thermal conductivity, \(\alpha_s\) is the substrate thermal diffusivity, \(t_{int}\) is the laser beam/substrate interaction time, \(m_p\) is the powder particle mass, \(C_p\) is the thermal heat capacity of powder material, \(T_{mp}\) is particle melting temperature, \(T_{ip}\) is particle initial temperature, \(A_s\) is the substrate absorption coefficient, \(A_p\) is particle absorption coefficient, \(A_{pe}\) is the powder cladding efficiency coefficient, \(S_{int}\) is laser beam/substrate interaction area size, \(\varepsilon\) is the powder extinction coefficient \(\varepsilon = 3(1-A_p)/2\rho_p r_p\), \(\rho_p\) is the powder material density,
$r_p$ is radius of powder particle, $v_p$ is the powder particle velocity and $S_p$ is the particle cross-section.

Solving the integral in Eqs. (3.5) and (3.6) numerically, we calculate $P_{ms}$ and $P_{mp}$ for all our laser cladding experiments, using mainly the input parameters from [23]. These calculations revealed an important role of the coaxial laser cladding parameter, namely the velocity of powder particles, $v_p$. This velocity is mainly determined by an amount of carrier gas used in the powder feeding system. The upper limit of this velocity can be estimated as the speed of the carrier gas escaping from the powder nozzle (0.25 – 1.5 m/s for our nozzle geometry).

Figure 3.24 clearly shows the crucial role of the powder particle velocity in the processing zone. The laser power required to melt the substrate as well as the clad particles is plotted as a function of particle velocity in the case of cladding 60µm size powder on steel substrate. Thermal properties of the powder can be found in [14], of substrate material as well as absorption coefficients used in this calculation can be found in [12]. The laser defocus distance, $L_d$, was +15 mm, the working distance $N_d$ was 14 mm and the powder efficiency coefficient $A_{pe}$ was set to 0.3 for this calculation, which represents a mean value observed in our experiments. As Figure 3.24 clearly demonstrates, the powder shielding effect increases $P_{ms}$ only at low particles velocities. When the particles velocity is higher than 0.5 m/s the $P_{ms}$ change with $v_p$ is not significant. On the other hand, the power required for heating the powder particle to the melting temperature $P_{mp}$ increases linearly with particles velocity. The intersection of these two graphs forms four different areas for the coaxial cladding regime as indicated in Figure 3.25. Inside the area I no cladding occurs. Inside the area II the clad track can be formed but the substrate is not melted and therefore no good metallurgical bond is formed between the laser track and the substrate. Laser cladding is not successful in this area. Somewhere inside the area III the laser cladding process lies in an optimal zone. Area IV may be characterized as an area of low efficiency laser cladding. Solid powder particles reach the liquid surface with relatively high velocities, which leads to the solid-particle/liquid-surface interaction with a high repelling force [18].

The velocity of clad particles was not varied in our experiments. However, the relatively steep dependence of $P_{mp}$ on particle velocity is a good assumption that for efficient coaxial cladding an optimal particle speed can be found for a wide interval of powder sizes and properties.

![Figure 3.24: Calculated laser power required to melt the substrate $P_{ms}$ and the particle $P_{mp}$ as a function of powder velocity for coaxial cladding of 19E Metco powder (80 µm size) on C45 steel substrate. Feeding rate $F = 141.7$ mg/s and scanning speed $S = 4.67$ mm/s.](image-url)
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3.3.2 Experimental on coaxial cladding process

In our experimental investigations of the coaxial laser process the nickel-chromium based alloy powder 19E Sulzer-Metco with mean particle size of 60 µm and the 60 mm diameter bar of C45 low-alloyed steel as a substrate were used. Chemical compositions of both materials are summarized in Table 3.2. To study the effects of the principal laser cladding parameters on the tracks we used a graded experiment [25]. This approach is based on a continuous increase of one process parameter while the others are kept fixed. Single clad track experiments were performed at all combinations of five different levels of powder feeding rates: \(F = 66.7, 91.7, 116.7, 141.7\) and 166.7 mg/s and five scanning speeds: \(S = 1.67, 2.67, 3.67, 4.67\) and 5.67 mm/s realized through the rotation of substrate bar under coaxial laser head. In each single track the laser beam power \(P\) increased continuously from 200 to 1500 W along one revolution of substrate bar. In brief, 25 laser tracks were performed to explore the whole processing window.

Computer controlled coaxial laser cladding system consists of: Metco 9MP powder feeder, coaxial cladding nozzle, XYZ-Rotation CNC table and the 2kW continuous wave Rofin Sinar Nd:YAG laser. The laser beam with top-hat energy distribution characteristic was focused 15 mm above the substrate resulting in laser beam spot size of 3.2 mm in diameter. The computer control allows operating the laser power \(P\) during the laser cladding via a linear change of laser lamps pumping power inside an interval 200-1750 W.

Because we study the laser cladding process using a laser power gradient experiment, the laser power gradient has to be “reasonably small”. The linear change of laser power along a single laser track results in laser power gradient of 6.9 W/mm. The change of the laser power inside a processing area is then smaller than 25 W, which seems to be a reasonably small value.

Table 3.2: Chemical composition of clad powder and steel substrate

<table>
<thead>
<tr>
<th>Material</th>
<th>Form</th>
<th>Element (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Ni  Cr  Si  B  Fe  Mn  Co  Mo  W  C</td>
</tr>
<tr>
<td>19E</td>
<td>Powder</td>
<td>Bal  16  4   4   4   -   2.4   2.4   2.4   0.5</td>
</tr>
<tr>
<td>C45</td>
<td>Bar</td>
<td>-    0.20 0.20 - Bal. 0.06 - 0.05 - 0.46</td>
</tr>
</tbody>
</table>

Figure 3.25 shows a typical cross section of one laser track and defines the main geometrical quantities usually used for laser track characterization: clad height \(H\) (mm), clad width \(W\) (mm), clad area \(A_c\) (mm\(^2\)) and molten area \(A_m\) (mm\(^2\)). From these characteristics two important quantities may be evaluated: dilution \(D\) and clad angle \(\alpha\). The dilution quantifies the relative amount of the substrate material that has been molten during the cladding process and mixed with an additional material, is given as:

\[
D = \frac{A_m}{(A_c + A_m)}. \tag{3.7}
\]
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Although for a good metallurgical bond some dilution between the coating and substrate is always required, the fact that the high dilutions may degrade coating properties mean that it stays reasonable low. In practice the clad angle $\alpha$ is required to be large enough to avoid porosity creation in the coating, while overlapping individual clad tracks. To avoid the systematic errors and considerable scatter of data an assumption is often made that the cross section of the laser track lies on a circle [26]. The clad angle $\alpha$ can be calculated from laser track width $W$ and laser clad height $H$ as [27]:

$$\alpha = 180 - 2\arctan\left(\frac{2H}{W}\right).$$

Due to the round shape of substrate, some geometrical features of laser tracks were measured using a non-destructive method. A profilometer was used to project the profiles of the laser tracks on a screen so that the height, the clad area and the width of the tracks could be calculated from digital images, Figure 3.26, using image analysis software.

![Figure 3.25: A typical laser track cross-section with its main geometric characteristics: clad height $H$, clad width $W$, clad area $A_c$, molten area $A_m$ and clad angle $\alpha$.](image)

![Figure 3.26: Non-destructive profilometer image of laser tracks, which can be used for quick evaluation of laser track geometric characteristics.](image)

This method is interesting when one needs quick method to evaluate laser clad characteristics. However, the study of internal features of the clad track, like the shape of the molten zone and the dilution still requires a cross sectioning. For recording of all geometrical features of the single tracks, clad rings were cross-sectioned at regular 45 degrees intervals. The samples were mechanically polished and chemically etched for metallographic observations. A corresponding value of the energy of laser beam at each sectioned surface was assigned using a known value of linearly increased pumping power and a laser calibration curve [28], which relates the pumping power and the laser beam output power. As Figure 3.27
clearly demonstrates all 25 laser tracks were cross-sectioned in 4 cuts, which provided a total of 175 usable data points corresponding to different combinations of S, F and P values.

Figure 3.27: A) 60 mm diameter C45 bar with laser tracks clustered in groups of five tracks with the same feeding rate, F. Scanning speed, S, and F increase from the left to right. The black pen lines mark the place with laser power of 362.5 and 1500 W. Detached laser tracks as well as segments without cladding are visible. B) Part of the experimental bar after the cutting for laser tracks inspection.

The laser cladding process is based on the heat transfer from the laser beam, the substrate and the powder and mass transfer between the powder flow and the molten surface. Therefore, the investigation of the process can be performed based on so called combined parameters [29]. Two quantities are fundamental. The amount of powder provided per unit length of the laser track $F/S$ and the total heat input per unit length of the laser track $P/S$.

Figure 3.28 brings the laser track cross-section map varying $P$ and $F/S$. This map demonstrates clearly some relevant aspects of the process parameters on the single clad track geometry.

First the robustness of the process is observed, which produces the laser track inside a very wide interval of laser cladding parameters. For instance, the clad area $A_c$ is changed from 0.2 mm$^2$ for the most delicate laser track located at the left upper corner to the more than 4 mm$^2$ for the largest one at the right down corner in Figure 3.28. Such an increase cannot be simply explained by the increase of the amount of clad material delivered per unit length $F/S$, because this quantity increases only 8 times. A substantial increase of powder-clad efficiency has to be concluded. Comparing the behavior of the clad height $H$ in the column marked as 45 g/m and in the row 720 W, one may conclude that the clad height $H$ mainly depends on $F/S$ parameter, while the laser power has a small impact on this quantity. On the other hand, the clad width is changed along the both axis directions in Figure 3.28. Cladding profiles above the already mentioned diagonal in Figure 3.28 are characterized by a small melting zone $A_m$ and therefore also by a low value of dilution $D$. At higher values of $F/S$ and low laser powers no good bond is created that leads to detaching of clad track from the substrate. In these cases the attenuation of the heat input into the substrate, due to powder shielding effect and high speed of the substrate, is so strong that the cladding process is realized under conditions described in area $\text{II}$ in Figure 3.24.

Further detailed observation presented in Figure 3.28 leads to the conclusion that the depths of the substrate melt area are mainly determined by the laser power $P$. Below the
diagonal in Figure 3.28 a characteristic shape of the substrate melt area for coaxial cladding is visible. Although a top-hat distribution of the laser energy inside a beam was used, the depth of the molten area strongly increases in the central part of laser track. An explanation for this behavior follows from a shape of powder cloud and from the powder distribution inside the processing zone. Of course, this distribution is not homogenous as it was for simplicity assumed in the model in Figure 3.24. In fact, the powder starts to get irradiated by the laser at the point of laser highest power density since its actual distribution is Gaussian. However, the powder shielding effect there is also the most effective, once the powder particles density at this distance from the substrate is the highest. In compensation to that, a part of laser energy stripped by powder shielding at this place is transported (in the form of thermal energy) to the central part of the laser track, where the powder stream is routed. This specific shape of the substrate melt area indicates that coaxial cladding set-up can be more susceptible to the formation of inter-run porosity due to shading of the laser beam, which is required to re-melt part of previous laser track.

![Figure 3.28](image_url) Laser tracks cross-section map for coaxial laser cladding of Ni-based powder on C45 steel substrate. Combined cladding parameter F/S on the horizontal axis characterizes the amount of fed material per unit length. Laser power given on vertical axis.

3.3.3 Process map of coaxial cladding

In order to quantify the above-mentioned qualitative observations the dependence of laser track characteristics on the main coaxial laser cladding parameters were investigated. We would like to note here that there were only small differences between the results obtained from non-destructive measurements of clad height $H$, clad width $W$ and clad area $A_c$ and the same values obtained during laser tracks cross-sections measurements. Figure 3.29 displays the dependence of the laser track height $H$ on the processing parameters. Figure 3.29A shows almost a linear increase of the clad height with increasing feeding rate F for different scanning
speeds $S$ at one laser power $P = 718\ W$. Laser clad heights close to 2 mm can be easily achieved choosing higher feeding rates and slower scanning speeds. Of course, some points with the highest values of $F$ and the lowest values of $S$ are missing in Figure 3.29A because of the strong powder shielding effect, as mentioned before. A detailed statistical analysis revealed that the clad height $H$ depends mainly on the feeding rate $F$ and on the scanning speed $S$. The laser power does not play an important role.

From a practical point of view it can be very useful if a correlation between an individual characteristic and one “combined process parameter” can be found. Usually, a “trial and error” method is used to find such a combined parameter. Statistical analysis is also able to estimate the importance of individual laser cladding parameter and/or their mutual interactions. As Figure 3.29B shows, the statistical linear correlation between laser clad height $H$ and the $F/S$ parameter exists with a relatively high regression coefficient $R=0.96$. A 95% confidence interval for linear regression is also shown. This result differs from the finding by Felde at al [30], which correlates the clad height to the $\sqrt{P.F/S^2}$ combined parameter with a similar regression coefficient. However, their laser cladding experiment was performed using the side cladding set-up, when the powder spent a shorter time inside the laser beam before it reached the melt pool. In such a set-up, the asymmetrical temperature distribution of the powder particles as well as an attenuation of the laser beam usually exists [31]. This may be the reason why the laser energy per unit length, $P/S$, is still an important parameter for the clad height. In coaxial cladding the clad height $H$ linearly increases with the amount of fed material per unit length.

Figure 3.29: A) Laser clad height $H$ as a function of powder feeding rate $F$ and scanning speed $S$ for fixed laser power $P=718\ W$. B) Laser clad height dependence on the combined parameter $F/S$. Dashed lines mark 95% confidence interval.
Figure 3.30 illustrates the behavior of laser track width, $W$, as a function of the process parameters. Now, the laser power $P$ and the scanning speed $S$ are shown to be crucial and the feeding rate $F$ seems to be the least important parameter. Figure 3.30A shows the dependence of the width of laser track $W$ on the laser power $P$, for different values of scanning speeds at one feeding rate $F = 91.7$ mg/s. The width of the laser track varies from 1 mm, observed at lower laser powers, to the values close to 3 mm, observed for a high laser power and low scanning speeds. The detailed statistical analysis also revealed, that at a constant laser power the laser track width $W$ decreases linearly with increasing scanning speed, as observed in a side cladding set-up [26,31]. However, the steepness of this decrease depends on the laser power. The combined parameter, which statistically correlates with the laser track width $W$, for the whole group of experimental points, is $P/\sqrt{S}$, as is clearly shown in Figure 3.30B. It is interesting to note, that it has been shown experimentally [26], and also in some model calculations [32,33], that the depth of the substrate melting zone increases linearly with laser power and that it is inversely proportional to the square root of scanning speed.

Combined parameters with relative high values of correlation coefficient (>0.95) were found for all directly measured quantities: $H$, $W$, $A_c$ and $A_m$. Clad angle $\alpha$, dilution $D$, and powder efficiency $P_e$ shown somewhat lower correlations with their combined parameters. Table 3.3 summarizes the combined parameters with the best correlations found with observed geometrical laser track characteristics. The powder efficiency $P_e$, was simply calculated from the measured clad area $A_c$, feeding rate $F$, scanning speed $S$, and powder density $\rho_p$ as:

$$P_e = A_c S \rho_p / F.$$  

As expected, the clad area $A_c$, strongly depends on amount of fed material per unit length $F/S$. This parameter quantifies the amount of powder, which builds the laser track. At higher power levels, almost all of the powder particles in the processing zone are melted and routed close to the melt pool center with a distribution, which can be described as Gaussian [33].
should be noted here, that we do not observed any change in slope of linear dependencies of $A_c$ or $P_e$ on laser power $P$. This was observed and explained in [25] as due to the power threshold at which powder is directly melted by the laser beam. This fact may be explained through the Figure 3.24. When the velocity of clad particles is selected in the interval where only a small difference between $P_{ms}$ and $P_{mp}$ exists, it is almost impossible to register two different cladding regimes on $A_c(P)$ dependence. The lower melting point of our powder material (1040 ºC) also suggests that we are working in such a regime.

In contrast, the molten area $A_m$ is mainly proportional to the laser power $P$, while the feeding rate $F$ and the scanning speed $S$ are parameters with less influence. In our experiments the value of clad angle $\alpha$ varies from 50 to 130 degrees. It simply shows to be proportional to the scanning speed $S$ and inversely proportional to the feeding rate $F$. Dilution $D$ varies from almost 0 to 55%. It is quite difficult to achieve a low dilution combined with a high laser clad height, mainly because of already mentioned specific shape of the molten area. Dilution of a single laser track from 10 to 30% has to be selected to form a thick and pore free coating by overlapping of individual laser tracks. However, the final dilution for a coating formed by overlapping can be expected to be smaller [29], because part of the beam energy is consumed in order to melt the overlapped track.

Finally, the powder efficiency factor $P_e$ is controlled by the combined parameter $P\sqrt{F}/S$. The value of $P_e$ is changed from approximately 15% observed at lowest values of the combined parameter, up to 50%, for its highest values. Almost the same correlation coefficient (0.90) was found for $P_e$ and $P/\sqrt{S}$ correlation. The combined parameter $P/\sqrt{S}$ describes the laser track width behavior. Therefore a correlation between powder efficiency and laser track width can be expected and this is confirmed in Figure 3.31. This correlation supports the assumption made in powder catchments models [29], namely that both, solid-liquid and liquid-liquid powder/substrate interactions lead to particle intercept during coaxial cladding. A further powder efficiency improvement is probably possible, by tuning the

**Table 3.3.: Combined parameters that shown the high correlations with geometrical characteristics of laser tracks. $R$ is the correlation coefficient, $a$ and $b$ are constants from the linear relation: $Q = a + bC$**

<table>
<thead>
<tr>
<th>Quantity $Q$</th>
<th>Combined parameter $C$</th>
<th>$R$</th>
<th>$a$</th>
<th>$b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H$ [mm]</td>
<td>$F/S$ [g/m]</td>
<td>0.97</td>
<td>0.164</td>
<td>0.023</td>
</tr>
<tr>
<td>$W$ [mm]</td>
<td>$P/\sqrt{S}$ [W s$^{1/2}$/mm$^{1/2}$]</td>
<td>0.95</td>
<td>0.947</td>
<td>0.002</td>
</tr>
<tr>
<td>$A_c$ [mm$^2$]</td>
<td>$\sqrt{PF/S}$ [W$^{1/2}$ g/m]</td>
<td>0.99</td>
<td>-0.2782</td>
<td>0.0019</td>
</tr>
<tr>
<td>$A_m$ [mm$^2$]</td>
<td>$P/\sqrt{FS}$ [W/(mg$^{1/3}$mm$^{1/3}$s$^{2/3}$)]</td>
<td>0.95</td>
<td>-0.3269</td>
<td>0.0073</td>
</tr>
<tr>
<td>$\alpha$ [degree]</td>
<td>$S/F$ [m/g]</td>
<td>0.90</td>
<td>55.0</td>
<td>961</td>
</tr>
<tr>
<td>$D$ [%]</td>
<td>$\sqrt{PS/F}$ [W$^{1/2}$ m$^{1/2}$/g$^{1/2}$]</td>
<td>0.92</td>
<td>-19.3</td>
<td>8.5</td>
</tr>
<tr>
<td>$P_e$ [%]</td>
<td>$P\sqrt{F/S}$ [W g$^{1/2}$/m$^{1/2}$]</td>
<td>0.91</td>
<td>14.2</td>
<td>0.0043</td>
</tr>
</tbody>
</table>
amount of carrier gas with the aim to minimize the particles velocity together with a stable powder stream cone.

![Graph](image)

**Figure 3.31:** Correlation between the width of laser track and powder efficiency for coaxial laser cladding of Ni-based powder on C45 steel substrate. Dashed lines mark 95% confidence interval.

In practical terms to form a non-porous coating the single tracks are deposited side by side with an overlap to minimize the roughness caused by “hills and valleys” profile. Inter-run porosity can be formed due to an acute clad angle. In order to form a dense layer, the angle $\alpha$ is suggested to be higher than 100° [30]. The second crucial condition for a strong coating is to keep the dilution at a reasonably low value. **Figure 3.32** represents the processing parameters map, which takes these two conditions into account with $S/F$ combine parameter on horizontal axis and $P$ parameter on vertical one. The points in the graph represent all combinations of experimental conditions used in our measurements. A condition for clad angle is represented by the vertical line made at $S/F = 0.047$ calculated from statistical dependence of clad angle on $S/F$ combined parameter. All points on the right side of this line fulfill the condition: $\alpha > 100^\circ$. As the dilution $D$ is linearly controlled by the combined parameter $\sqrt{PS/F}$, the constant dilution curve will form a hyperbolic function in **Figure 3.32**. Curves for constant dilutions of 5 and 30 % are depicted by two solid curves. The experimental area limited by these two solid curves and by the vertical line from a left side reveals the practical processing window (shaded area in **Figure 3.32**) desirable for the production of non-porous, low diluted coatings. If the vertical line, which indicates a condition for clad angle, seems to be a considerable limiting factor, a change of the laser beam spot size may be the solution, which allows forming a coating at higher laser power once a flatter clad track can be produced. Statistical relation between clad track height $H$ and $S/F$ allows also drawing of second horizontal axis with reciprocal scale for laser track heights. Finally, the linear relation between the clad area $A_c$ and $\sqrt{PF/S}$ allows to mark six dashed isolines for $A_c$ from 0.25 to 5 mm$^2$. **Figure 3.32** thus summarizes the entire set of experiments, together with some statistical relations between laser track characteristics and coaxial laser cladding parameters. It can be used as a guide for selecting processing parameters selection for a required coating.
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Figure 3.32: Processing window for coaxial laser cladding in P vs S/F representation. Points correspond to analyzed laser track cross-sections. Vertical solid line determines the clad angle condition required for continuous coating; two solid hyperbolas terminate an area of allowed dilution and the grey area shows the window, where an optimal clad layer can be formed. Dashed curves are isolines with denoted values of clad area $A_c$.

3.4 Analysis of side cladding process

3.4.1 Experimental on side cladding process

For the analysis of the side cladding processing window, the commercial Co-based alloy powder Eutroloy 16006 from Castolin Eutectic, see Table 4.1, with the mean particle size of 140 µm and compositionally close to widely used Stellite 6 alloy was used for laser cladding on a gray cast iron flat substrate. The side laser cladding geometry [34] was used in powder feeding system consisting from: Metco Twin 10C powder feeder, argon as carrier and shielding gas and ALOtec Dresden GmbH Cu-based side cladding nozzle with a cyclone and 2 mm nozzle opening. All laser cladding experiments are carried out with the already described 2 kW Nd:YAG laser system (Rofin Sinar CW20). CNC (4 axes) table was used to move the laser beam and the powder feeding system over the substrate with a controlled scanning speed. All lengths and areas quantities were measured from digital photographs of the cross-sections following the same procedure applied for the coaxial setup. It has been shown already that a systematic investigation of the laser cladding process can be based on so-called laser processing combined parameters. Figure 3.33 represents the laser track cross-section visual map for $P$ and $F/S$ variations. Again, the robustness of the process is a
remarkable feature. When a low laser power is combined with a high amount of clad powder per unit length, then almost all laser power is absorbed by the powder particles and no melt pool can be created in the substrate. Therefore no clad tracks are formed. On the other side, when small amount of the powder is clad together with a high laser power, non desired amount of dilution (usually connected with a degradation of coating properties) occurs. From the observations of the laser track cross-sections along the descending diagonal of Figure 3.25 a significant increase of clad area $A_c$ has to be concluded. This increase cannot be fully explained just by an increase of delivered powder per unit length. The combined parameter $F/S$ roughly triples by an increase from 17 to 50 g/m while the clad area increases much more than three times. Therefore, a substantial increase in powder efficiency has to be expected. Similar prediction can be made about the dependency of clad height, when observing this quantity along the columns of Figure 3.33. It seems that the clad height $H$ does not change significantly with increasing laser power $P$, when $F/S$ parameter is constant. In order to quantify these qualitative observations, the dependence of laser track characteristics on the main laser cladding parameters were analyzed statistically.

![Figure 3.33: Laser tracks cross-section map for side cladding of Co-based alloy on grey cast iron substrate. Combined parameter F/S on the horizontal axis characterizes the amount of fed material per unit track length.](image)

**3.4.2 Process map of side cladding**

The statistical analyses for all defined laser clad track quantities (track width $W$, clad area $A_c$, molten area $A_m$, clad angle $\alpha$, dilution $D$ and powder efficiency $P_e$) were performed in a similar procedure as for the coaxial process. Table 3.4 summarizes all combined parameters with the best correlations found with observed geometrical laser track characteristics for side laser cladding of Eutroloy 16006 powder on gray cast iron substrate and compares them with
parameters found in the work for coaxial cladding and in work of Felde et al. [30] for side cladding of Stellite6 on C45 steel substrate.

It is interesting to note, that the combined parameters found for laser clad height $H$, laser clad width $W$ and clad area $A_c$ are the same as in the case of coaxial laser cladding. Although the regression coefficient for $W$ is relatively low ($R=0.9$), we like this parameter $P/\sqrt{S}$ for its simplicity and consistency with coaxial laser cladding case. For the molten area $A_m$ a little bit simpler parameter $P^2/\sqrt{S}$ was found in comparison with coaxial cladding. It depends only on two main laser cladding parameters with a correlation coefficient exhibiting even higher value (0.98 against 0.95). However, the combined parameters for the rest of laser track characteristics: clad angle $\alpha$, dilution $D$ and powder efficiency $P_e$ were found in more complicated forms, in comparison with parameters found for coaxial cladding. It seems, that the simple laser track characteristics as laser track height $H$, laser track width $W$ and clad area $A_c$ are controlled by the same combined parameters as for coaxial cladding. The way of powder delivery (coaxial or side) does not play an important role for these characteristics. A more complicated situation is observed for the rest of laser track characteristics, probably because more interactions and physical phenomena are involved. It is interesting to note, that for coaxial cladding the highest value of powder efficiency was observed of about 50 %, but for side cladding set-up the maximal value of this quantity approaches 90 %. This is due to a better focusing of powder stream to the processing molten area and due to the entrapment of particles on the corner formed between clad track and substrate.

Table 3.4: Combined parameters that shown high correlations with geometrical characteristics of laser tracks for side laser cladding set-up and their corresponding regression coefficients. Combined parameters found for coaxial cladding set-up by Oliveira et al [34] (* marks the same combined parameter as in column 2) and for side cladding set-up by Felde et al [30].

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Combined parameter</th>
<th>Coaxial cladding [34]</th>
<th>Side cladding [30]</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H$</td>
<td>$F/S$</td>
<td>0.97 *</td>
<td>$\sqrt{P.F}/S^2$</td>
</tr>
<tr>
<td>$W$</td>
<td>$P/\sqrt{S}$</td>
<td>0.90 *</td>
<td>-</td>
</tr>
<tr>
<td>$A_c$</td>
<td>$\sqrt{P.F}/S$</td>
<td>0.98 *</td>
<td>-</td>
</tr>
<tr>
<td>$A_m$</td>
<td>$P^2/\sqrt{S}$</td>
<td>0.98 $P/\sqrt{F.S}$</td>
<td>-</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>$F/\sqrt{P.S}$</td>
<td>0.94 $S/F$</td>
<td>$S/F$</td>
</tr>
<tr>
<td>$D$</td>
<td>$\ln(P/F.\sqrt{S})$</td>
<td>0.94 $\sqrt{P.S}/F$</td>
<td>$P.S/F$</td>
</tr>
<tr>
<td>$P_e$</td>
<td>$\ln(P^2/\sqrt{F})$</td>
<td>0.94 $P.\sqrt{F}/S$</td>
<td>-</td>
</tr>
</tbody>
</table>

In the application of laser cladding process the coating is formed by an overlapping of single laser tracks deposited side by side with a displacement, which minimizes the roughness

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caused by “hills and valleys” profile. In practice a displacement of 2/3 of single laser track width is often used. Such an overlapping creates the coating, which is usually 20-35 % thicker than the height of single laser track. The dilution observed for the coating is reduced due to the fact that during the laser cladding of overlapped laser track a part of the laser beam energy is consumed by remelting of a side of clad material in previous laser track [13]. In our experiments we used 33 % overlapping of the laser tracks and we found, that the dilution drops from its original value observed in the first laser track to a new “stable” value after three clad tracks. This new reduced value of dilution depends mainly on a used laser power and varies between 30-60 % of the original value observed in the first laser track.

On the base of this knowledge we are able to construct the processing parameters map for the side cladding set up, relating some geometrical properties of cladding with their processing parameters, as it was designed for coaxial laser cladding set-up. Figure 3.34 represents such map for the side laser cladding of Co-based powder on grey cast iron substrate with $S/F$ parameter on horizontal and $P$ parameter on vertical axis, respectively. The rectangular points inside the map show all combinations of experimental parameters used in our measurement. Because of linear statistical relation between $H$ and $F/S$, the second horizontal axis for $H$ may be drawn and clad height may be directly deducted for each combination of experimental conditions. It should be noticed here, that this height $H$ represents the height of individual laser track and not the final thickness of the coating, which is always slightly thicker depending on the overlapping factor used. The linear relation between $A_c$ and the combined parameter $\sqrt{P.F} / S$ allows marking the dashed isolines, which connect the experimental points with the same value of clad areas $A_c$. To incorporate also the clad angle and the dilution onto our laser cladding map a small modification of best combined parameters presented for these quantities in Table 3.4 has to be made. For the clad angle $\alpha$ combined parameter $S/F$, which is preferred for coaxial and other side cladding set-up, is more appropriate to incorporate this quantity into Figure 3.34. However, relatively low correlation coefficient (only $R=0.86$) was calculated for this complex parameter. Therefore the vertical line on the processing map marks only approximately a limit for dangerous $\alpha$\textdegree <100\textdegree from the point of view of inter-run porosity [17]. As the second combined parameter for dilution in Table 3.4 also contains $S/F$ parameter, curves for constant dilution two solid-hyperbola like curves, i.e. $D = 15$ and 45 % are depicted. The area between these two lines represents the processing window of acceptable dilutions for laser clad coatings (~5-25 %). Together with the condition for clad angle and with respect to the experimentally explored area, the space of processing parameters for a successful formation of laser coating may be marked by a cross-hatched area in Figure 3.34.

3.4.3 Application of a cladding processing map

Laser processing maps should be used as a guide for the first “guessing” and subsequent “tuning” of optimal laser processing parameters during laser cladding of continuous coatings. As an example, the Figure 3.35 shows the cross-sections of Co-based coating prepared by side laser cladding process on compacted graphite iron substrates. The coating with thickness of about 1 mm may be created in a single clad layer without any macro-pores and cracks.
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Laser cladding in second and third layer allows forming of thicker coatings, as Figure 3.35B, C also demonstrates. However, when thicker coatings (>2 mm) were formed on a relatively stiff substrate (as the plate on Figure 3.35E) a cracking inside the coating or in the substrate (initiated from a side of coating/substrate interface) were sometimes observed. Figure 3.35 also shows a possible geometrical variability during overlapping of laser tracks. Squared coated areas were formed via overlapping of individual lineal laser tracks (Figure 3.35E) and circular coatings result from a continuous spiral cladding (Figure 3.35D).

Figure 3.34: Laser processing map for side laser cladding of Co-based alloy powder on grey cast iron substrate in P vs. S/F representation. Data points indicate values of processing parameters of analyzed laser track cross sections. Vertical solid line corresponds to the condition for clad angle required for continuous coating. Two solid hyperbolic type lines mark out the area of acceptable dilution and crosshatched area shows the window, where an optimal clad layer can be formed. Dashed curves are isolines with denoted values of clad area $A_c$. 
3.5 Conclusions

Laser Hardening.

The possibility of temperature controlled laser surface hardening with a 2 kW Nd:YAG laser beam on pearlitic gray cast iron was investigated aiming to improve its surface quality. After hardening of GG-25 cast iron by Nd-YAG laser beam with scan speeds from 2-50 mm/s there are several different microstructural regions found in the transformed zone. The transformed zone mainly consists of martensite, retained austenite, remaining cementite lamellae and undissolved graphite flakes. Hardened depths~ 300-700 µm are obtained.

Observation of remaining cementite lamellae and ghost pearlite indicate that a pearlite/austenite transformation at high heating rates takes place according to the direct mechanism: $\text{Fe}_\alpha (+ \text{Fe}_3\text{C}) \rightarrow \text{Fe}_\gamma (+ \text{Fe}_3\text{C})$.

Micro hardness has increased by the laser treatment from 250 HV of the base material to 500-1000 HV in the transformed zone. The micro hardness depth profile corresponds to the change in microstructure.

Coaxial cladding process.

Theoretical and experimental study of the coaxial laser cladding of Ni-based powder on steel substrate revealed several specific features that distinguish the coaxial cladding from the traditional side-cladding set-up.

The model is based on the geometry of the coaxial laser head, which provides the minimal laser power for co-melting the substrate and powder particles. The calculations predict a crucial role for the velocity of powder particles in the coaxial powder stream. This velocity may be used as another process parameter for fine-tuning of the whole process.

The experimental set-up, in which the laser power is gradually changed while other two main process parameters are fixed, allows a substantial reduction of laser cladding
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experiments required in order to properly define the processing window. Empirical relations between the basic laser track characteristics and the main process parameters were found for all studied laser track characteristics with a correlation coefficient higher than 0.9. Some of these relations differ from relations observed for a standard side-cladding set-up.

The rotational symmetry of the powder/laser beam interaction zone and the cone shape of powder flow determine the specific shape of the substrate melt area, which is deeper in its central part in comparison with a substrate melt area formed during a side cladding set-up. Therefore, a higher value of dilution may be required in order to form the laser track with good bonding.

Side cladding process.

The experimental study of laser cladding of Co-based powder on two cast iron substrates in traditional side-cladding set-up and subsequent study of geometry, microstructure and properties allow the following conclusions.

The grade experiment set-up, in which the Nd:YAG laser power is gradually increased during laser cladding of a single laser track while other two main process parameters (scanning speed and powder feeding rate) are fixed, contributes to a substantial reduction of laser cladding experiments usually required for a proper study of the whole processing window.

A wide processing window is observed for side-cladding of Stellite-6 type powder on cast iron substrates. Empirical statistical relations between the single laser track characteristics and the main process parameters were found for all studied characteristics with correlation coefficients higher than 0.9. Some of these relations differ from relations observed for coaxial laser cladding. The processing map for laser cladding of Eutroloy 16006 Co-based alloy powder on cast iron substrates was designed on the base of these empirical relations.
References

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