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Development and characterization of nickel based tungsten carbide laser cladded coatings

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Abstract

Laser cladded coatings consisting of various types of tungsten carbides embedded in a NiCrBSiCFe matrix are characterized. At optimal process parameters crack-free coatings with a thickness of 0.85-1 mm, excellent bonding with the substrate, carbide concentrations up to 60 wt% and a hardness in the range of 40-55 HRC are obtained. During laser cladding the carbides have partly dissolved in the matrix as indicated by the presence of dispersed carbides in the matrix and by a carbide phase growing into the matrix along the edges of the particles. The wear coefficient during sliding contact decreases logarithmically with increasing carbide concentration.

Keywords: Processes; laser cladding; metal matrix composite; material properties; wear

1. Introduction

Friction and wear of machine components result in great loss of energy and materials. Surface technologies, such as thermal spraying, laser cladding and arc deposition welding are able to produce thick wear resistant coatings on the surface of metal components. One approach to improve the wear properties of a metal surface involves the addition of hard particles (such as carbides, borides, nitrides and oxides) to it. Metal matrix composites (MMC) consist of a combination of hard ceramic particles such as carbides embedded in a tough metallic binder. Laser cladding is capable of producing a wide range of MMC coatings. During lasercladding a laser source melts the substrate and metal-ceramic composite powder is injected simultaneously into the molten pool. The metal powder with relatively low melting point melts and acts as a binder for the ceramic phase. Thanks to the superficial melting of the substrate, a strong metallurgical bond is formed between substrate and coating. The high cooling rate results in a fine microstructure and minimal reaction between the ceramic and metallic phase. Some degree of reaction between the ceramic and metallic phase is reported to be beneficial for wear resistance as this improves the bonding between the ceramic phase and the metallic matrix. Tungsten carbide is often used as ceramic strengthening phase in laser cladded MMC coatings thanks to its combination of high hardness, certain plasticity, good wettability by molten metals and low thermal expansion [1].

The matrix material is usually a nickel or cobalt based alloy.

The structure of laser cladded nickel alloy – tungsten carbide powders has been investigated in several studies. The degree of dissolution depends on the laser beam – composite interaction time, carbide particle size and volume fraction of carbides [1]. Dissolution of WC particles in the matrix leads to secondary carbides dispersed in the matrix and around the primary carbides [2]. In laser cladded NiCr - spherical WC coatings following carbide phases were distinguished by Amado et al.: WC, W,C, WC, WC/ [3]. Other authors report in addition mixed carbide phases such as Ni2W4C [4],[5], M,C [6] or FeW,C, Fe,W,C, (W,Cr,Ni)2,C [7].

The abrasive wear resistance of Ni based coatings containing different types of tungsten carbides was studied in [2], [4], [8]. The effect of spherical tungsten carbide size and volume fraction on both sliding and abrasive wear of laser cladded Ni based
tungsten MMC coatings was studied by Van Acker et al. [9]. It was observed that smaller particles were more effective in reducing wear. A logarithmic decrease of wear coefficient with volume fraction of carbides was reported.

In the present investigation, MMC coatings consisting of various concentrations of tungsten carbide particles in a nickel based matrix are produced by laser cladding. Spherical and angular shaped tungsten carbide powders produced by different powder manufacturers are used as feedstock. The coatings are characterized in terms of microstructure, hardness and sliding wear resistance.

2. Experimental procedure

2.1. Feedstock powder

As metal matrix material a nickel based alloy with following chemical composition was selected: Ni (bal.) – 7.5Cr – 1.6B – 3.5Si – 0.3C – 2.6Fe. The nickel alloy has self fluxing properties, which is favourable for thermal coating processes.

As strengthening phase, various tungsten carbide powders with roughly the same particle size and spherical or irregular shape have been used (Table 1).

Tungsten carbide powders and nickel based powders are supplied in a ratio ranging from 0-60wt% during laser cladding.

<table>
<thead>
<tr>
<th>Designation</th>
<th>Supplier</th>
<th>Shape</th>
<th>Particle size</th>
</tr>
</thead>
<tbody>
<tr>
<td>S_WOKA</td>
<td>Sulzer Metco</td>
<td>Spherical</td>
<td>45-106 μm</td>
</tr>
<tr>
<td>S_Hoganas</td>
<td>Hoganas</td>
<td>Spherical</td>
<td>45-106 μm</td>
</tr>
<tr>
<td>I_Hoganas</td>
<td>Hoganas</td>
<td>Irregular</td>
<td>45-125 μm</td>
</tr>
</tbody>
</table>

2.2. Laser cladding setup

Laser cladding experiments are performed with a fiber-coupled 3 kW Laserline diode laser and coaxial cladding nozzle. The laser spot diameter at the substrate is 3.75 mm (1.5 mm fiber diameter; collimator with 100 mm focal length; focal lens with 250 mm focal length). Argon gas is used as transport gas for the powder and as shielding gas in the coaxial cladding nozzle. The Ni based and WC powders are not premixed but supplied using two separate hoppers to avoid segregation. The optimal process parameters (laser power, scan speed, preheating temperature, powder flow,...) have been determined from analysis of the coating properties such as thickness, dilution, cracks, pores, distribution of carbides,... A coating thickness after laser cladding in the range of 0.85-1 mm was required. The same process conditions were applied for the 3 different carbide powders at equal carbide concentrations. For the MMC coatings, preheating of the substrate was required to avoid cracks in the coating. At a higher carbide concentration the use of a higher preheat temperature and lower laser power was optimal, as shown in Table 2. Coatings are produced on substrates of SAE8620 steel (55 mm diameter, 10 mm thickness).

<table>
<thead>
<tr>
<th>Coating composition</th>
<th>Scan spacing (mm)</th>
<th>Scan speed (mm/min)</th>
<th>Laser power (W)</th>
<th>Powder mass flow rate [Ni / WC] (g/min)</th>
<th>Preheating temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni 1540</td>
<td>1.5</td>
<td>500</td>
<td>1000</td>
<td>8.5 / 0</td>
<td>/</td>
</tr>
<tr>
<td>Ni 1540 + 20% WC</td>
<td>1.5</td>
<td>500</td>
<td>850</td>
<td>8.3 / 2.1</td>
<td>160</td>
</tr>
<tr>
<td>Ni 1540 + 40% WC</td>
<td>1.5</td>
<td>500</td>
<td>800</td>
<td>7.4 / 4.9</td>
<td>180</td>
</tr>
<tr>
<td>Ni 1540 + 60% WC</td>
<td>1.5</td>
<td>500</td>
<td>750</td>
<td>6.1 / 9</td>
<td>200</td>
</tr>
</tbody>
</table>
2.3. Characterisation

The laser cladded coatings are characterized in terms of microstructure, hardness and phase constitution. The results obtained by optical microscopy, Electron Probe X-ray Micro-Analysis (EPMA), X-Ray Diffraction (XRD) and hardness testing are presented in section 3.

Tribological tests are performed using a Wazau TRM 1000 tribometer to evaluate and compare the wear resistance of the coatings under sliding conditions. For each coating structure at least two samples have been tested. The test conditions are as follows:

- Spherical Al₂O₃ counterbody with diameter of 6 mm
- Load = 150 N
- Temperature = 430°C
- Air atmosphere
- Radius of wear track = 16-18 mm
- Duration = 0.5 or 1 hour
- Laser cladded samples are tested in grinded and polished condition

For evaluating the wear resistance, profilometry of the wear track at 4 random positions along its circumference is performed. The average wear volume is used to calculate the wear coefficient, which expresses the wear volume per unit load and unit traversed distance. Scanning electron microscopy of the wear track is performed to analyse the wear mechanism.

3. Results and discussion

3.1. Analysis of tungsten carbide feedstock powders

The tungsten carbide powders (see Table 1) are analyzed in terms of structure and phase constitution by X-ray diffraction and microscopy. The results are summarized in Table 3. The X-ray diffraction profiles are given in Fig. 1. For all the powders a mixed phase constitution is revealed. The WC and W₂C phases are present in all the powders but not at the same concentration. The ratio of W₂C phase to WC phase is calculated for all the powders from the XRD profile using the Rietveld method. This ratio is for the S_WOKA powder significantly higher than for the other two powders. In relation to wear resistance it is important to note that the W₂C phase is harder but also more brittle than the WC phase. Furthermore, the W₂C phase dissolves more easily in the metallic matrix during laser cladding than the WC phase. A higher degree of tungsten carbide dissolution in the metal matrix is harmful for two reasons: it leads to a reduction in toughness of the matrix and consequently a high cracking susceptibility during cladding and it leads to a loss of the beneficial property of the ceramic as strengthening phase. The S_WOKA powder contains, besides of the WC and W₂C phases, also the WC₁₋ₓ phase. The WC₁₋ₓ phase is not present in the S_Hoganas powder but instead XRD peaks that can be attributed to tungsten and graphite are detected. The S_Hoganas powder is known to be produced by casting. For the angular powder only the WC and W₂C phases are resolved. This phase constitution is present in tungsten carbide powders produced by eutectic melting of liquid carbon and tungsten, followed by conventional casting and crushing to the desired shape (‘fused’ powder).

![Fig. 1. X-ray diffraction profiles of tungsten carbide powders (see Table 1)](image-url)
Table 3. Properties of tungsten carbide powders applied as feedstock powder (see Table 1)

<table>
<thead>
<tr>
<th>Designation</th>
<th>Hardness (HV0.5)</th>
<th>Phases present</th>
<th>W_2C / WC</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>I_Hoganas</td>
<td>2600 ± 500</td>
<td>W_2C, WC</td>
<td>63% / 37%</td>
<td>Dense</td>
</tr>
<tr>
<td>S_Hoganas</td>
<td>3480 ± 150</td>
<td>W_2C, WC, W, C</td>
<td>60% / 40%</td>
<td>Dense</td>
</tr>
<tr>
<td>S_WOKA</td>
<td>4700 ± 800</td>
<td>W_2C, WC, WC_1-x</td>
<td>85% / 15%</td>
<td>Spherical pores</td>
</tr>
</tbody>
</table>

An important quality difference between the spherical powders is revealed by optical microscopy of the powders after polishing (see Fig. 2). The S\_WOKA powder contains particles with spherical pores while the cast S\_Hoganas is fully dense.

![Optical micrographs of spherical tungsten carbide powders](image)

(a) S\_WOKA  
(b) S\_Hoganas

Fig. 2. Optical micrographs of spherical tungsten carbide powders

3.2. Structural analysis of laser cladded coatings

Crack-free coatings with excellent bonding to the substrate and a thickness of 0.85-1 mm could be successfully produced by laser cladding using all carbide types at carbide concentrations up to 60wt%. The analysis results are illustrated below for the coatings containing 60wt\% tungsten carbide. Depending on the process parameters, a homogeneous distribution of carbides in the coating is obtained. In general, a lower concentration of carbides is present near the position of the overlap. The coating produced with S\_WOKA powder contains the highest amount of spherical pores, mainly located inside the carbide particles (Fig. 3(a)). This is not surprising as the initial powder contains pores (see Fig. 2(a)), which cannot escape from the coating as the carbides remain mainly undissolved during cladding. A small quantity of spherical pores is also present inside the matrix for all coatings (Fig. 3).

![Micrographs of laser cladded coatings](image)

(a) Ni-alloy + S\_WOKA  
(b) Ni-alloy + S\_Hoganas  
(c) Ni-alloy + I\_Hoganas

Fig. 3. Micrographs of laser cladded coatings produced using Ni alloy + 60wt\% irregular and spherical tungsten carbides at optimal process conditions.
The XRD profiles of the coatings produced with 60 wt% tungsten carbide powders are compared in Fig. 4. The main peak corresponds to an austenitic nickel based phase γ-Ni. In the Ni alloy coating without carbide addition the presence of a Ni rich boride phase of the type M3B is detected but in the MMC coatings this is less clearly resolved due to overlap with diffraction peaks from carbide phases. The carbide phases that are clearly present in all the coatings are W2C and WC. The W2C/WC ratio is smaller in the coatings than in the powders (see Table 3 and Fig. 4). This has also been reported in literature and attributed to the lower thermal stability of W2C compared to WC leading to preferential dissolution of W2C [8]. A large reduction in W2C/WC ratio is observed for the coatings produced with the angular powder (I_Hoganas) and the S_WOKA powder. For these coatings the presence of mixed carbides (type (NiWFeC)xC) is clearly observed by XRD (see Fig. 4), while this is less obvious for the S_Hoganas powder. Angular carbides have in general a higher tendency to dissolve than spherical ones due to the presence of sharp corner, which might be an explanation for the lower stability of the I_Hoganas powder compared to the S_Hoganas powder [8].

![X-ray diffraction profiles of laser cladded coatings produced using Ni alloy + 60wt% irregular or spherical tungsten carbides. The type of tungsten carbide powder applied is indicated (see Table 1).](image)

The presence of mixed carbides (grey small dispersed phase in Fig. 5) in the metal matrix (black phase in Fig. 5) is confirmed by Electron Probe Micro Analysis. The micrographs show that the particles have partly dissociated at their edges and a carbide phase growing into the metal matrix is formed around them. As reported before some dissolution of the carbides at their edges is beneficial as this tends to improve the bonding between the ceramic phase and the metal matrix and reduces hence the tendency to pull-out the tungsten carbide particles during tribological contact. The structure inside the angular carbide powder is clearly resolved by EPMA and consists of WC needles in a W2C matrix. The structure inside the spherical fused carbide powder (S_Hoganas) is at these magnifications not resolved, which is probably caused by the small scale of the structure formed by rapid cooling during powder production.

![EPMA micrographs of coatings produced by laser cladding using Ni alloy + 60wt% irregular or spherical tungsten carbides](image)

### 3.3. Evaluation of hardness and wear resistance of laser cladded coatings

Because of the bad quality in terms of porosity of the spherical carbides from Sulzer Metco (S_WOKA), no further evaluation of these coatings in terms of wear resistance is performed. Fig. 6(a) shows the wear coefficient of the coatings as a function of weight percentage tungsten carbide used in the feedstock powder. The wear decreases logarithmically with increasing carbide
content for both carbide types. There is no clear trend showing which carbide type results in the highest wear resistance during the applied contact conditions in the full concentration range studied. The spherical carbides exhibit a lower wear rate at carbide concentrations of 20wt% and 40wt% while the opposite is observed at 60wt%. The wear coefficient of the MMC coatings as a function of their Rockwell hardness is displayed in Fig. 6(b). At the same feedstock concentration the coatings with spherical carbides tend to have a lower hardness compared to the ones with irregular carbides while the opposite trend applies when comparing the tungsten carbide feedstock powders (see Table 1). However, the difference is small taken into account the spread in hardness values.

Fig. 6. Wear coefficient of laser cladded coatings produced using Ni alloy as matrix material and various amounts of spherical and angular tungsten carbides as a function of (a) the tungsten carbide concentration in the feedstock powder and (b) the Rockwell hardness of the coating. The test conditions during tribotesting are described in section 2.2.

Fig. 7 shows the scanning electron micrographs of the wear track for the coatings produced with spherical and irregular carbides. The wear mechanism is essentially the same for both type of coatings. The width of the wear track is of the order of 1 mm. Preferential wear of the softer matrix occurs. Scratches are mainly formed in the matrix but occasionally some fine scratches run through the carbides. In most cases, the scratches in the matrix stop or bend off when a carbide particle is reached (see top micrograph in Fig. 7(a)). In front of the primary carbides pile-up of worn off material occurs. This results from preferential wear of the matrix and the accompanying height difference between the primary carbides and the matrix. No pull outs of carbides are observed but occasionally complete fragmentation of primary carbides occurs. At the downstream side of the primary carbides cracking inside the carbides occurs preferentially (see bottom micrograph in Fig. 7(a)). The wear debris are fine
(order of 1 μm) and indicated by white arrows in the top micrograph of Fig. 7(b). Energy-dispersive X-ray spectroscopy reveals that these debris are oxides, which contain some aluminium from the alumina counterbody and a relatively large amount of tungsten. This indicates that the debris not only result from wear of the matrix but also of the tungsten carbides, which have reacted with oxygen to form hard (mixed) oxides.

4. Conclusions

- Crack-free nickel based tungsten carbide coatings with a carbide concentration up to 60wt% have been produced by laser cladding. A coating thickness of 0.8-1 mm has been obtained after one single pass.
- Spherical and irregular tungsten carbide powders from different suppliers have been characterized. A difference in porosity and phase constitution (WC, W₂C, WC₁₋ₓ, graphite and W) was obtained.
- At optimal laser cladding parameters, some dissolution of the tungsten carbide particles in the nickel based matrix occurs leading to fine dispersed mixed carbides in the metal matrix and a carbide phase growing into the metal matrix around the primary carbides.
- The wear coefficient during sliding contact with an Al₂O₃ counterbody at elevated temperature decreases logarithmically with volume fraction of carbides for the spherical and irregular carbides. The wear mechanism is for the coatings containing angular and spherical tungsten carbides essentially the same. Preferential wear of the matrix occurs. Abrasive wear debris, which are characterized as being mixed oxides, cause scratches mainly inside the matrix. There is no clear trend showing which carbide type is most beneficial at the applied contact conditions for the full concentration range.

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References