Metal Matrix Composite Material by Direct Metal Deposition

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Abstract

Direct Metal Deposition (DMD) is a laser cladding process for producing a protective coating on the surface of a metallic part or manufacturing layer-by-layer parts in a single-step process. The objective of this work is to demonstrate the possibility to create carbide-reinforced metal matrix composite objects. Powders of steel 16NCD13 with different volume contents of titanium carbide are tested. On the base of statistical analysis, a laser cladding processing map is constructed. Relationships between the different content of titanium carbide in a powder mixture and the material microstructure are found. Mechanism of formation of various precipitated titanium carbides is investigated.

Keywords: rapid manufacturing; laser cladding; direct metal deposition; metal matrix composite; titanium carbide

1. Introduction

Direct Metal Deposition (DMD) is a laser cladding method that allows fabricating near-net-shape metal parts from a CAD solid model in one step [1, 2]. Laser cladding is a method of depositing material by which a powdered material is melted and consolidated by use of a high power laser [3]. The DMD technology can reduce the overall part production time and cost [4, 5].

Metal Matrix Composite (MMC) denotes a class of composites with at least two constituent materials one of which is a metal [6]. MMCs are applied in automotive and aerospace industry owing to enhanced high temperature strength, fatigue resistance, wear resistance and lightweight design [7]. MMCs are produced by laser cladding from a wide range of alloys and particulate reinforcement phases. Performance characteristics of a MMC object are influenced by the properties of the particulate reinforcement phase such as chemical composition, shape and size, properties as ingredient material, volume fraction and spatial distribution in the matrix [8]. Various studies have confirmed the technological advantages of the laser powder cladding over the conventional deposition welding in the field of composite materials [9].

The objective of this research is to demonstrate the possibility to produce by DMD carbide-reinforced MMC objects from a mixture of high-strength steel 16NCD13 and titanium carbide powders. Regression analysis of the influence of the laser cladding process parameters on the track geometry is carried out. A laser cladding process map

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Table 1. Input process parameters $P$, $S$ and $F$ for the laser cladding of steel/titanium carbide powder mixture.

| Input parameters | 1  | 2  | 3  | 4  |
|------------------|----|----|----|----|
| Laser power $P$  | kW | 2.6| 3.2| 3.8| 4.4|
| Scanning speed $S$ | m/min | 0.6| 0.7| 0.8| 0.9|
| Power feeding rate $F$ | Tr/min (g/min) | 2800 (20.1) | 3600 (26.4) | 4400 (32) | 5200 (36.6) |

Table 2. Coefficients of the regression equations for the geometrical characteristics of an individual track.

| Output parameter | $P$ | $S$ | $F$ | $P \cdot S$ | $S \cdot F$ | $P \cdot F$ | $P^2$ | $S^2$ | $F^2$ |
|------------------|-----|-----|-----|-------------|-------------|------------|------|------|------|
| Height $H$ mm    | $\beta_0$ | $\beta_1$ | $\beta_2$ | $\beta_3$ | $\beta_4$ | $\beta_5$ | $\beta_6$ | $\beta_7$ | $\beta_8$ | $\beta_9$ |
| Width $W$ mm     | 3.584 | 0.681 | -0.249 | -0.143 | -0.059 | -0.137 | 0.070 |
| Depth $h$ mm     | 0.158 | 0.042 | -0.012 | -0.012 | -0.028 | -0.007 | -0.022 | -0.008 |

for manufacturing MMC material is established. The process parameters are optimized to fabricate MMC objects with high deposition efficiency. Different types of MMC structure with precipitated TiC particles are studied.

2. Experimental procedure

2.1. Materials

Matrix Metal Composite material was manufactured on cast iron substrate S235 from a mixture of commercially available powders: (a) high-strength low-carbon steel 16NCD13 (Fe3Ni1CrMo) by Sandvick Osprey Ltd. (Neath, UK) with -106+45 µm particle size; (b) titanium carbide TiC by Testbourne (Hampshire, UK) with -80+40 µm particle size. The powders were premixed in different proportions: 85 and 15; 90 and 10; 95 and 5; 97.5 vol% of steel and 2.5 vol% of titanium carbide, respectively.

2.2. Experimental setup

The experiments were performed on Trumpf DMD 505 commercial industrial-scale laser cladding installation equipped by a 5 kW continuous wave CO₂ laser system. A laser spot of 5 mm in diameter with a TEM₀₁⁺ energy distribution is formed on the substrate at 20 mm distance from a nozzle tip. A powder cladding set-up consists of: computer-controlled powder feeding system, coaxial cladding nozzle mounted on CNC five-axis gantry. Coaxial powder injection is realized by nozzle, carrier, shaping gas mixture of Ar and He. Experiments were performed at gas flow rates $G_{\text{carrier}}$ (Ar/He) = 18/2 l/min, $G_{\text{shaping}}$ (Ar) = 10 l/min, and $G_{\text{nozzle}}$ (Ar/He) = 15/1 l/min.

2.3. Experimental plan

To analyze the influence of the main laser cladding parameter on the geometry of an individual track, an experiment plan was established (Table 1). In total, $n = 64$ tracks of 25 mm length were cladded. The main track geometrical characteristics such as track height $H$, track width $W$ and substrate melting depth $h$ were measured in the beginning, the middle and the end of each laser track, and then their average values were calculated. The technological characteristics such as dilution $D = h/(H + h)$ and powder deposition efficiency $E_p = 2/3 \cdot p \cdot H \cdot W \cdot S/F$ were estimated. Layers were produced by overlapping individual laser tracks by 3 mm in each step. Multilayer objects of 35x35x12 mm³ size were fabricated by criss-cross manufacturing strategies i.e. the cladding directions of two consecutive layers were perpendicular to each other.

After etching by HCl/FeCl₃ solution, cross sections of the multi-layered object samples were subjected to microstructure and chemical composition analysis on a scanning electron microscope TESCAN Vega 3 SB with EDS. The Vickers microhardness testing was performed on BUEHLER Omnimet MHT 5104 equipment.
3. Results and discussion

3.1. Laser cladding process map

In statistics, regression analysis includes any techniques for modeling and analyzing several variables, when the focus is on the relationship between a dependent (or output) parameters \( y \) and independent (input) parameters \( x \). A regression model relates \( y \) to a function of \( x \): \( y = f(x) \) \[10\].

In this study, the laser cladding parameters such as laser power \( P \), scanning speed \( S \), power feeding rate \( F \) were input parameters, the geometrical characteristics such as track height \( H \), track width \( W \), and substrate melting depth \( h \) were output parameters. To evaluate the level of influence of the laser cladding parameters on the track geometry, values of input parameters were normalized. In our case, the mathematical description of the model is presented in the form of a polynomial of the second degree (Table 2).

Given a data set \( \{y_i, x_{i1}, \ldots, x_{ip}\} \) of \( n \) statistical units, a nonlinear regression model takes form

\[ y_i = \beta_0 + \beta_1 x_{i1} + \ldots + \beta_p x_{ip}, \quad i = 1, \ldots, n \] \[1\]

Often these \( n \) equations are combined together and written in the vector form as

\[ Y = \beta X \] \[2\]

Then the final solution is written as

\[ \beta = (X^T X)^{-1} X^T Y \] \[3\]

After the computation, the regression model for each output parameter has been constructed (Table 2). In order to assess the goodness-of-fit of the model, the R-squared, analyses of the pattern of residuals and hypothesis were used. Statistical significance was checked by an F-test of the overall fit followed by t-tests of the individual input parameters [11].

Table 2 summarizes all the found regression coefficients for the geometrical characteristics for the track laser cladding from the steel/TiC powder mixture. It should be noted that the laser power \( P \), the scanning speed \( S \) and the powder feeding rate \( F \) have almost the same effect on the track height \( H \). For the track width \( W \), the major parameter is the laser power \( P \). Under conditions applied in this study, a variation of the powder feeding rate \( F \) does not lead to a significant change of the diameter of the powder flow in the working area. But a two-time rise of the laser power \( P \) from 2.5 up to 5 kW leads to the increasing from 3.8 to 5 mm of the spot diameter \( d_{0.86} \), and, thus, of the size of
On the base of the present statistical analyze, a laser cladding process map was depicted (Figure 1). The dashed curves present three different levels of dilution \(D\) of 15, 25, and 35%, respectively. The hyperbolic solid curves correspond to diverse track heights \(H\) changing from 0.2 up to 0.7 mm. Different powder deposition efficiency \(E_p\) is given by the dotted curves. The zone of acceptable dilution and high powder deposition efficiency is cross-hatched. Further experiments were realized with these optimal parameters.

The laser cladding process map also allows estimating the layer thickness which usually exceeds by 10-30% the track height and depends on the step between the overlapping individual tracks.

### 3.2. Phase diagram of system Fe-TiC

16NCD13 powder is a low-carbon low-alloy steel. To estimate at a first approximation the effect of titanium carbides TiC on the microstructure and the properties of the steel, an iron alloy with TiC will be considered (Figure 2) [12].

The quasi-binary section Fe-TiC has a eutectic-type phase diagram with 3.8 wt% of TiC content in the eutectic. The maximum solubility of TiC in Fe is 0.6 wt%. Concentration of the elements dissolved in a solid solution increases with the cooling rate relatively to the equilibrium crystallization. These solid solutions are called metastable or supersaturated. During laser cladding, high cooling rates (> 1000 K/s) contributes to the formation of a supersaturated solid solution of TiC in \(\gamma\)-phase [13]. In this case, crystallization takes place according to the metastable phase diagram (Figure 2).

To determine the influence of TiC on the microstructure and properties of the steel, the following contents of TiC in the steel were selected: 2.5 vol% (1.6 wt%) (hypoeutectic alloy); 5 vol% (3.2 wt%) (near-eutectic alloy), and 10 vol% (6.4 wt%) (hypereutectic alloy).

### 3.3. Hypereutectic alloy

The hypereutectic alloy containing 10 vol% (6.5 wt%) of TiC consists of three phases: 1 – primary titanium carbide TiC\(_{prim}\); 2 – eutectic \(E (\gamma + TiC)\); 3 – secondary titanium carbide TiC\(_{second}\) (Figure 3).

TiC\(_{prim}\) nucleated in the form of compact polyhedrons (small-scale dendrites) which become centers of the crystallization of the eutectic.

The crystallization of the eutectic \(E (\gamma + TiC)\) is not equilibrium at high cooling rate which is characteristic for laser cladding. After the crystallization of TiC\(_{prim}\), the liquid phase is depleted by titanium carbide near TiC\(_{prim}\) crystals. TiC in \(\gamma\)-phase has not time to completely diffuse because of a high cooling rate. Therefore, in the beginning the eutectic crystallizes in the form of a rim from \(\gamma\)-phase around TiC\(_{prim}\) crystals. Then, the eutectic...
composition aligns, and the eutectic assumes common lamellar and rod-like structure (Figure 3).

Secondary titanium carbides TiC\textsubscript{second} precipitate in the eutectic phase due to a change in the solubility of TiC in the $\gamma$-phase from 0.6 up to 0.011 wt% in the solid state. However, these precipitations are difficult to identify even at a large variation of the solubility because their shape is very similar to one of the eutectic.

MEB analysis of the chemical elements distribution confirmed the hypothesis of the structure formation. The concentration of the titanium is increased in the primary titanium carbides and the eutectic colonies. The other elements are uniformly distributed in the alloy.

### 3.4. Hypoeutectic alloy

The hypoeutectic alloy with 2.5 vol% (1.6 wt%) TiC content consists of two phases: 1 – supersaturated solid solution TiC in $\gamma$-Fe; 2 – excessive titanium carbides TiC (Figure 4).

The solubility limit of TiC in $\gamma$-Fe increases because of a high value of the supercooling, and the alloy turns to be $\gamma$-monophasic. A supersaturated solid solution of TiC in $\gamma$-Fe forms. The excessive TiC precipitates at the grain boundaries because of the repeated heating of the metal by the upper layers (Figure 4).

The analysis of the distribution of the chemical elements showed a high concentration of titanium on the grain boundary in the region of the excessive TiC precipitation. The other elements in the alloy are distributed uniformly.

### 3.5. Near-eutectic alloy

The structure of the near-eutectic alloy with 5 vol% (3.2 wt%) TiC content consists of two phases: 1 – supersaturated solid solution TiC in $\gamma$-Fe; 2 – eutectic $E$ ($\gamma$ + TiC) (Figure 5).

The supersaturated solid solution of TiC in $\gamma$-Fe forms in the same way as in the alloy with 2.5 vol% TiC content. But the alloy is outside the $\gamma$ monophasic region because of a higher TiC content. As a result, eutectic colonies in the lamellar and rod-like form precipitate at the dendrite boundaries (Figure 5). The precipitation of TiC\textsubscript{second} does not occur due to the formation of supersaturated solid solution of TiC in $\gamma$-Fe.

MEB analysis revealed a high concentration of titanium in the region of eutectic colonies. The other elements of the alloy are homogeneously distributed.
3.6. Hardness

The hardness tests of the MMC material yielded highly diversified results which indicated a considerable influence of the type of structure on the properties of the samples (Figure 6).

At the 2.5 and 5 vol% TiC content, the hypoeutectic alloy has a considerable hardness that is higher by 40% in the middle and by 90% in the upper layers than that of the pure steel. The formation of the supersaturated solid solution of TiC in $\gamma$-Fe with a strong distortion of the crystal lattice increases sharply the hardness. The hardness of the hypoeutectic alloy reaches up to 550HV$_{0.1}$. But the hardness decreases up to 400HV$_{0.1}$ in the middle because of the repeated heating of the alloy by the upper layers. The precipitation of excessive TiC from the supersaturated solid solution of TiC in $\gamma$-Fe reduces the distortion of the crystal lattice.

The hardness of the hypereutectic alloy (10 vol% of TiC) insignificantly exceeds that of the pure laser-cladded steel. Nonequilibrium eutectic formed in the alloy is composed of the $\gamma$-phase in the form of a rim around TiC$_{prim}$ crystals, and the eutectic $E(\gamma + TiC)$. The microhardness of the $\gamma$-phase depleted by titanium carbide is lower than that of the eutectic. As the volume fraction of the $\gamma$-phase considerably exceeds the amount of the other phases, the alloy hardness is determined by a more plastic $\gamma$-phase.

4. Conclusions

A carbide-reinforced metal matrix composite material from a mixture of low-alloy steel 16NCD13 and titanium carbide TiC powders was produced by laser cladding.

Equations of relationship between the main laser cladding parameters and the geometrical characteristics of the cladded tracks were derived by regression analysis. On the base of statistical study, laser cladding process map for the deposition of individual tracks was established. Optimal process parameters with acceptable dilution and high efficiency for laser deposition of MMC material were established.

MMC material with 2.5, 5, and 10 vol% of TiC content fabricated by laser cladding presents three different structures, respectively:

1 – supersaturated solid solution TiC in $\gamma$-Fe and excessive titanium carbides TiC (hypoeutectic alloy);
2 – supersaturated solid solution TiC in $\gamma$-Fe and eutectic $E(\gamma + TiC)$ (near-eutectic alloy);
3 – primary titanium carbide TiC$_{prim}$; nonequilibrium eutectic $E(\gamma + TiC)$; secondary titanium carbide TiC$_{second}$ (hypereutectic alloy).

Alloys with the low TiC content (2.5 and 5 vol%) have the highest microhardness (about 550 HV$_{0.1}$) due to the formation of the supersaturated solid solution TiC in $\gamma$-Fe with a strong distortion of the crystal lattice. At the 10 vol% of TiC content, the material microhardness insignificantly exceeds that of the pure laser-cladded steel (about 280 HV$_{0.1}$) because of the nonequilibrium eutectic that is composed mainly of the ductile $\gamma$-phase in the form of a rim around the TiC$_{prim}$ crystals.

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