

## Laser remelting of Al-Fe-TiO powder composite on aluminium matrix

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### Manufacturing and processing

#### ABSTRACT

**Purpose:** Synthesis of the AlFeTiO<sub>3</sub> composite powder lead to formation metal matrix composite layer. The formation of an in-situ surface layer was carried out on aluminium alloy substrate using laser cladding technique.

**Design/methodology/approach:** It was assumed in the material and technological conception that the low laser energy initiates high exothermic synthesis reaction in the AlFeTiO<sub>3</sub>. This change the AlFeTiO<sub>3</sub> powder structure and intermetallic phases as well as aluminium oxide would be formed. The structure of the composite powders and the cross-section of the laser clad region were analyzed by optics and scanning microscopy, and X-ray microanalysis method.

**Findings:** The correlation of the microstructure of powders remelted with the aluminium substrate was determined, depending on the laser beam energy density. There were aluminium oxide and intermetallic phases from the Al-Fe-Ti system present in the remelting region.

**Research limitations/implications:** Laser parameters process such as density of laser energy as well as the time of interactions could be optimized to find the laser parameter for remelting of the AlFeTiO<sub>3</sub> composite.

**Originality/value:** A complex multiphase ceramic matrix composite microstructure was displayed consisting of intermetallic compound Al<sub>3</sub>Ti distribute in the Al<sub>2</sub>O<sub>3</sub> interdendritic region.

**Keywords:** Composites; Laser; Intermetallic phases

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### 1. Introduction

Limitations in use of aluminium alloys include wear, temperature and corrosion resistance considerations. Laser surface treatment (alloying/cladding) is a most promising innovative

technique among the various surface treatment process to overcome these problems. The laser high intensity beam is an ideal tool for the surface treatment techniques e.g. surface melting, alloying, cladding processes for improved corrosion, wear and microstructure refinement [1,2]. The basic laser surface treatment could be connected with the fast moving laser source in

order to obtain very high cooling speeds, in the order of  $10^6$  K/s, of the material subjected to treatment, which in consequence results in the formation of untypical structures (e.g. metallic glass) of the remelted material, impossible to be obtained by conventional methods [3-5].

In the material and technological conception presented herein it was assumed that the laser beam energy would initiate an exothermic process of melting of reactive composite powders located on an aluminium substrate. As a result of the exothermic reaction initiation by a laser beam [6,7], the additionally generated energy facilitates the remelting of the composite powder and the generation of a connection between the remelted composite powder and the aluminium matrix/substrate. The remelted composite powder constitutes a material of a new phase composition: an intermetallic phase's alloy with aluminium oxide with the eutectic structure [7-12]. Due to their physicochemical properties, intermetallic phase alloys reinforced with ceramic phases, belong to the group of advanced materials of a broadening range of applications [12,13].

This paper presents the results of research on two different laser technologies applied to melt a reactive composite powder with an aluminium matrix/substrate. The blown powder laser cladding method with a powder feeder: the composite particles (powder) are radiated by a laser beam, which is carried by gas flow and clad on the aluminium substrate immediately. The second method is direct laser cladding. The composite powder is pre-placed on the substrate material, the powder will be melted and clad on the substrate material when the laser scans across and through the cladding layer [14]. The purpose of the investigations conducted was to determine the technological parameters "window" of laser induced reaction synthesis and remelting of AlFeTiO<sub>3</sub> composite powders on an aluminium substrate. In this work the eutectic alloy of intermetallic phases with alumina will be expected to produce as a result of laser remelting.

## 2. Experiment

### 2.1. Composite material

AlFeTiO<sub>3</sub> composite powder with 70 vol.% of aluminium powder reinforced with 30 vol.% FeTiO<sub>3</sub> oxide particles were milled together by rotary-vibration mill. This technique is known in literature as a mechanical alloying or mechanical milling [15]. The exact technological parameters of mechanical alloying process had been determined in previous investigations [15,16]. The obtained agglomerates of the reinforcement particle of the AlFeTiO<sub>3</sub> composite show homogeneous distribution of ceramic particles throughout the whole volume of the aluminium matrix. Morphological and microstructural characterization of the AlFeTiO<sub>3</sub> agglomerates were carried out by Scanning Electron Microscope, (SEM), Hitachi -S3400N using the Energy Dispersive X-ray microanalysis (EDX). Figure 1 shows the morphologies of the AlFeTiO<sub>3</sub> particles.

The typical results of EDX analysis for AlFeTiO<sub>3</sub> composite grains before laser treated were presented in Table 1.

Table 1.

Chemical composition of the AlFeTiO<sub>3</sub> composite powder

Position at the Fig. 1 (down)	Al (Wt. %)	Ti (Wt. %)	Fe (Wt. %)
su1	3.72±0.39	42.66±0.27	51.27±0.94
su2	6.75±0.36	39.60±0.21	52.82±1.17
su3	13.79±0.34	36.39±0.24	48.41±0.96
su4	97.53±0.54	0.79±0.15	1.67±0.41
cs1	100±0.52	0	0
cs2	64.52±0.31	13.51±0.24	20.96±0.51

### 2.2. Laser processing - blown powder laser cladding method

In blown powder laser cladding method, the 10 mm thick AlSi-alloy substrate plate which is to be clad was moved at a constant velocity while the cladding AlFeTiO<sub>3</sub> composite powder was poured onto it using a pneumatic powder delivery system. The arrangement for cladding using this method is presented at Fig. 2.

For the experiment a continuous High Power Diode Laser (HPDL) ROFIN DL020 emitting optical radiation at a wavelength 940 nm was used. The lens system used to focus the HPDL laser beam onto the AlSi-alloy substrate work piece consisted in cylindrical lens with a focal length of 66 mm. The focused beam had a rectangular profile 1.8 mm × 3.8 mm which was traversed at 90° to the slow axis therefore producing 3.8 mm track along the AlSi-alloy substrate. The particles are injected into the melt pool at an angle of 60° with respect to the surface of the AlSi alloy substrate. AlFeTiO<sub>3</sub> composite powder is injected into the laser pool, just behind the beam in such way that the powder is positioned close to the axis beam with partially touching it. This caused that AlFeTiO<sub>3</sub> composite powder particles are preheated tentatively before they are deposited on the surface of the AlSi-alloy melted with a laser beam.

The key point for the realization of the laser cladding in this method is the shape of the melt pool which forms just below the laser beam. The shape of the thermal field in the XZ plane along the laser beam axis (Fig. 2) was determined based on the modified theoretical model [17].

$$T(\xi, r) = T_0 + \frac{AIS}{2\pi kr} \exp\left[-\frac{v}{2a}(\xi + r)\right] \quad (1)$$

where:  $r = \sqrt{\xi^2 + y^2 + z^2}$  coordinates of the field, and the thermophysical properties of AlSi-alloy substrate:  $k=159$  (W·m<sup>-1</sup>·K<sup>-1</sup>) – the thermal conductivity;  $\rho=2650$  kg·m<sup>-3</sup> the density;  $c_p=913$  (J·kg<sup>-1</sup>·K<sup>-1</sup>) - specific heat of the AlSi-alloy substrate material;  $a=(k/(\rho \cdot c_p))$  - the thermal diffusivity;  $T_0$  - the ambient room temperature of the experiment,  $I = P/S$  – the laser energy flux per unit area ( $S$  - the surface area of the laser spot beam).  $P$ - the total power of the laser beam,  $R=1-A$  the reflectance of the work piece,  $A$ - is the laser radiation absorptivity (defined as the ratio between the intensity absorbed by the work piece and the incident intensity),  $v$ - the relatively speed of the laser beam and work piece.

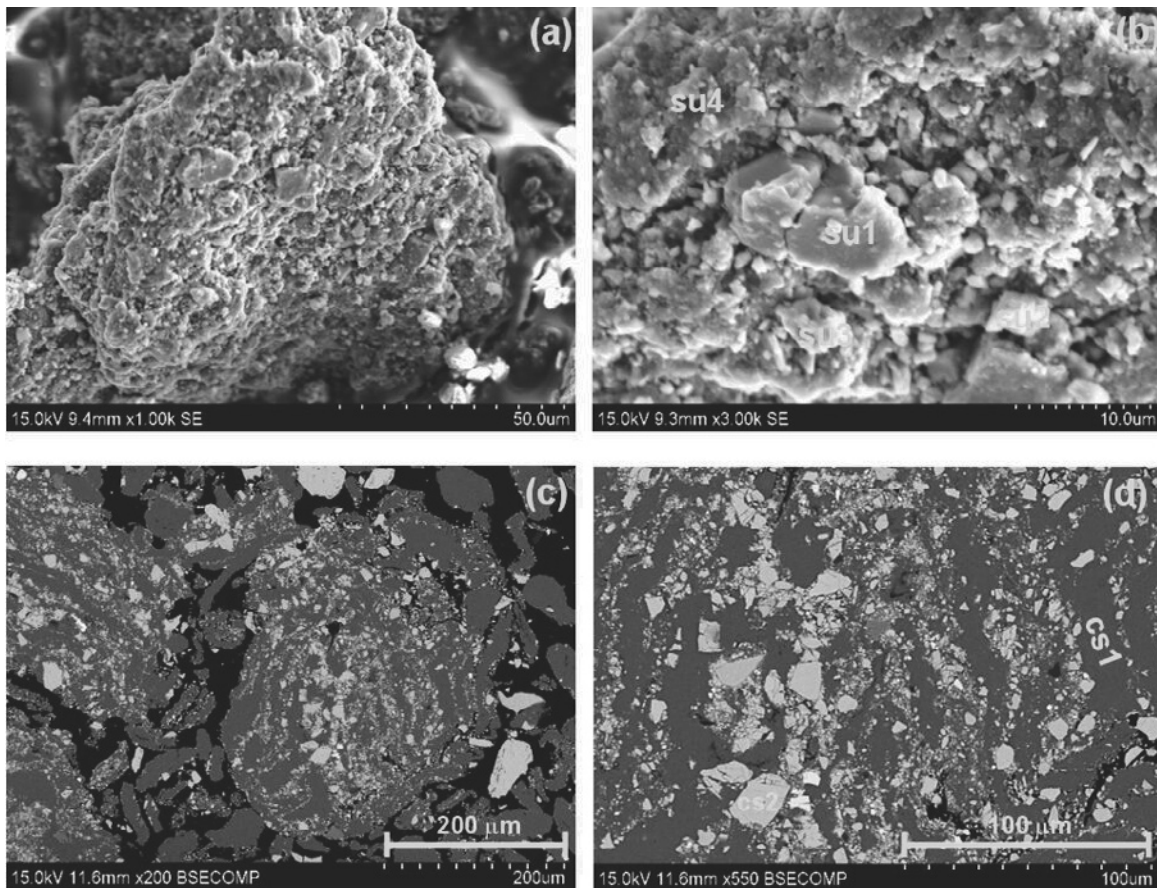


Fig. 1. The images of AlFeTiO<sub>3</sub> composite grain powder morphologies after mechanical alloying process, before laser treatment. (a,b) the surface of the grain; (c,d) the cross section of the grain

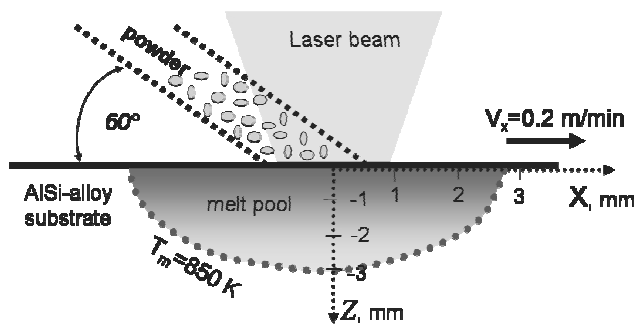


Fig. 2. The arrangements for laser cladding method with using blown AlFeTiO<sub>3</sub> composite powder. The shape of the melt pool for AlSi-alloy was obtained by the physical modeling. The constants of laser processing: the traverse speed of the AlSi-alloy substrate  $v_x=0.2$  m/min, HPDL laser power  $P=2200$  W with rectangular profile beam  $1.8$  mm  $\times$   $3.8$  mm

For the purpose of calculation of the maximum depth of the AlSi-alloy melting with a HPDL laser beam, the lumped

parameter model proposed by Kar [18] was applied. In this model the overall energy are considered and the following relation is deduced [18,19].

$$\frac{d}{P} = \frac{A}{a_o(vw + A_3\sqrt{vw})} \tag{2}$$

$$\text{where } A_3 = \left( \frac{wk + 2kl}{2a_o\sqrt{alw}} \right) (T_m - T_o) \tag{3}$$

$$\text{and } a_o = \rho [c_p(T_m - T_o) + L_m + \beta L_b] \tag{4}$$

where  $l=1.8$  mm is the length,  $w=3.8$  mm the width of the HPDL rectangular laser spot, and the other thermophysical properties of AlSi-alloy substrate are next:  $T_m=850$  K - the melting temperature,  $L_m=3.97 \cdot 10^5$  (J $\cdot$ kg<sup>-1</sup>), latent heat of melting,  $L_b=87 \cdot 10^5$  (J $\cdot$ kg<sup>-1</sup>) latent heat of evaporation and  $\beta$  is the contribution of evaporation of the AlSi-alloy surface.

In the theoretical calculations of this work, the experimentally absorptivity values of the AlSi-alloy was determined for the wavelength  $\lambda=940$  nm. This absorptivity depends on the alloy surface temperature and its course is presented in Fig. 3. The dependence of  $R(T)$  for metals results from the Drude free-electron theory and the theory of electron-phonon scattering. Because of increase in phonons numbers with the temperature, increases the electron-phonon collision frequency and in consequence increases absorptivity. In Fig. 6 is evident that for AlSi-alloy the reflectance decreases (absorptivity increases) at temperature above the Debye's temperature of AlSi-alloy substrate (for aluminium  $\theta_D=380$ K).

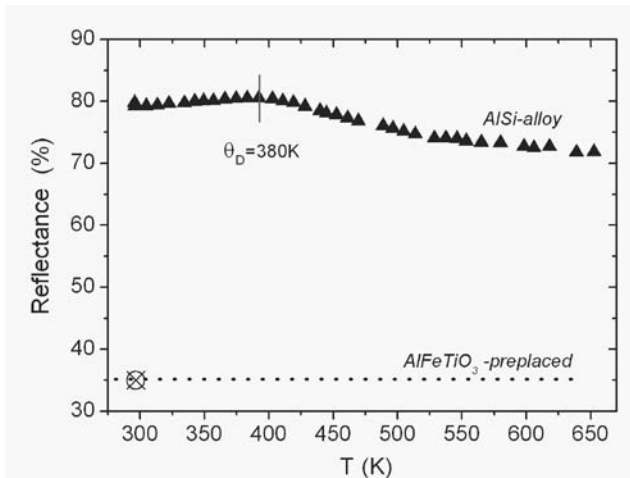


Fig. 3. Temperature dependence of the reflectance ( $R=1-A$ ) for the normal incidence of electromagnetic radiation. ▲- experimental data for AlSi-alloy substrate for  $\lambda=940$  nm, measurement was conducted in vacuum; dot line - experimental data for prep laced AlFeTiO<sub>3</sub> composite powder for  $\lambda=10.6$   $\mu$ m measured at constant temperature 297 K

The calculated from physical modeling the thermal field shape was compared with the experimental results. A comparison of the obtained experimental results with theoretical curves allowed the calibration of a relevant model and next, an extension of its application for other parameters of laser processing. The thermal field shaped determined for parameters applied in experiment is shown in Fig. 2. Interestingly enough, the feeding of powder (for powder feeding rate up to almost 9 g/min) does not change the thermal field distribution in any significant way.

**Laser processing - preplaced powder laser cladding method:**

In this laser cladding method the AlFeTiO<sub>3</sub> powder has been preplaced onto aluminium substrates. The substrate samples used were in the form of 150 mm x 65 mm sheet with a 5 mm thick (99.6 wt.%) purity aluminium.

The arrangements for laser cladding are shown in Fig. 4. The composite powder AlFeTiO<sub>3</sub> was pressed in the 60x2x2 grooves

on aluminium sheet under a pressure of 2.5 MPa. The single track cladding of AlFeTiO<sub>3</sub> powder was processed by CO<sub>2</sub> laser radiation under the protected of argon. A continuous 1.8 kW CO<sub>2</sub> laser was used for all experiments in this method. The CO<sub>2</sub> laser beam was defocused on the top surface of the work piece to a spot size of 2 mm.

**3. Result and discussion**

Figure 5 shows the typical surface appearance and the cross-section macrostructure of (metalloceramic) AlFeTiO<sub>3</sub> composite layer obtained by the blown powder laser cladding method. On the surface of the layer obtained, characteristic drops of the melted composite powder are visible (Fig. 5a). Their diameter is in the order of one millimeter, located in the middle of the layer, where the intensity of the HPDL laser beam was the greatest. The chemical composition of the drops is shown in Table 2.

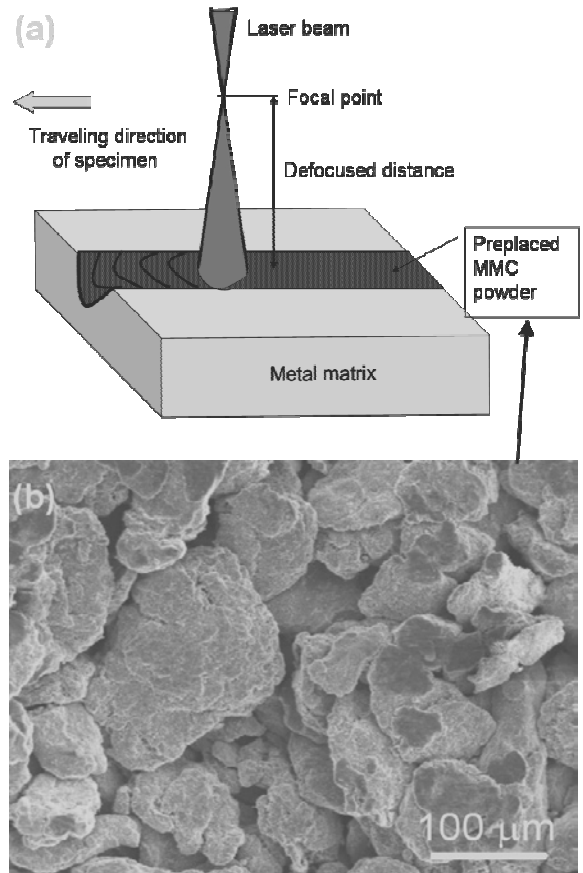


Fig. 4. The arrangements for laser cladding using preplaced AlFeTiO<sub>3</sub> composite powder (a). SEM microstructure of the original AlFeTiO<sub>3</sub> composite powder pressed powder before CO<sub>2</sub> laser treated (b)

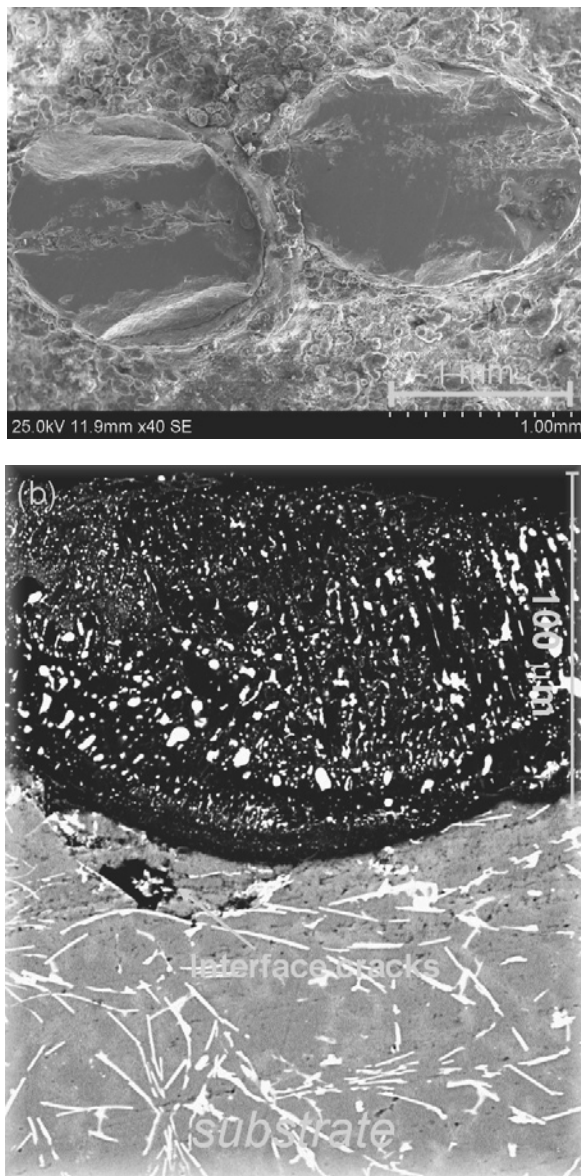


Fig. 5. The top view (a) and the cross-section (b) of HPDL laser cladding of the  $\text{AlFeTiO}_3$  composite powder onto AlSi-alloy substrate. Laser power  $P=2.2\text{ kW}$ , AlSi-alloy work piece traverse speed  $v=0.2\text{ m/min}$ . The powder feeding rate is constant, equal  $6\text{ g/min}$

The metalceramic layer of the  $\text{AlFeTiO}_3$  obtained on the surface of the substrate consisting of the AlSi-alloy is  $100\text{ }\mu\text{m}$  thick in its middle part. The average chemical composition of the layer is provided in Table 2. On the cross-section of the layer (Fig. 5b), a connection of the layer with the AlSi-alloy substrate is visible. The pores in the area of the layer/substrate connection are visible, too. In the middle part of the connections, a shrinkage cavity is visible. There are some cracks surrounding the cavity. The occurrence of the shrinkage cavity in the middle part of the connection is caused by a very large temperature gradient in that

place and by different coefficient of thermal expansion of the substrate material and metalceramic layer of the melted composite powder [20]. The obtained layer is uniform in its structure. However, single pores of a distinct vertical symmetry are observed in it.

In case of the other method presented in this work, the  $\text{CO}_2$  laser beam acts on the preplaced  $\text{Al-FeTiO}_3$  composite powder. The mechanism of the laser beam operation is a bit different here. The electromagnetic radiation emitted by a  $\text{CO}_2$  laser is strongly absorber by the preplaced  $\text{AlFeTiO}_3$  composite powder. During the first laser beam transition, ca. (80-70)% of the beam energy is absorbed in the surface layer of the preplaced composite powder, as shown in Fig. 3. Because of low thermal conductivity in the preplaced  $\text{AlFeTiO}_3$  composite powder in compare to the aluminium substrate/matrix the laser beam energy is not effectively transferred deep into the composite at the beginning of the laser treatment. However the laser beam initiates an exothermic reaction in the  $\text{AlFeTiO}_3$  composite material surface layer. During the synthesis of  $\text{Al}_3\text{Ti}$  and  $\text{Al}_2\text{O}_3$  in individual grains, a strong exothermic reaction, a lot of latent heat is released. The composite powder grain  $\text{AlFeTiO}_3$  begins to melt and a heterogenic mixture is formed. The intermetallic phases from Fe-Al and Ti-Al systems, aluminium oxide and the non-reacted aluminium matrix are synthesised in situ in the laser clad melt pool. The  $\text{AlFeTiO}_3$  remelting process with a high intensity laser beam moving along the treated surface at a constant speed, and with the initiated by the laser beam energy exothermic reactions, is a complex and dynamic process.

Fig. 6a,b,c shows the SEM microstructure in the cross-section of the composite cladding layer in the area of the laser beam action where the final effect is a fully remelted  $\text{AlFeTiO}_3$  composite powder. There is a eutectic alloy structure in its middle part. In Fig. 6b, typical effects of quick crystallization are visible, with columnar crystals ( $\text{Al}_2\text{O}_3$  dendrites) oriented so that their growth direction corresponds to the highest temperatures gradient direction [21]. From examining the fragment of composite material presented in Fig. 6 a,b it was found that the composite  $\text{AlFeTiO}_3$  components were completely remelted and blended together.

Comprehensive analysis by SEM and EDX (Table 2) shows the chemical composition of selected areas of an analysis of the structure formed.

Table 2. Chemical composition of the elements performed on lasers remelted  $\text{AlFeTiO}_3$  composite using EDX microanalysis method

position	Al (Wt %)	Ti (Wt%)	Fe (Wt%)
Analysis position at Fig. 5.			
top	42.44±0.29	25.09±0.22	31.72±0.44
cross section	74.12±0.25	22.62±0.21	69±0.20
Analysis position at Fig. 6.			
p1	81.33±0.39	18.69±0.27	0
p2	82.80±0.36	17.20±0.21	0
p3	75.23±0.32	10.20±0.13	14.57±0.22
p4	88.45±0.38	6.33±0.15	5.22±0.22
p5	76.69±0.32	9.34±0.13	13.97±0.22

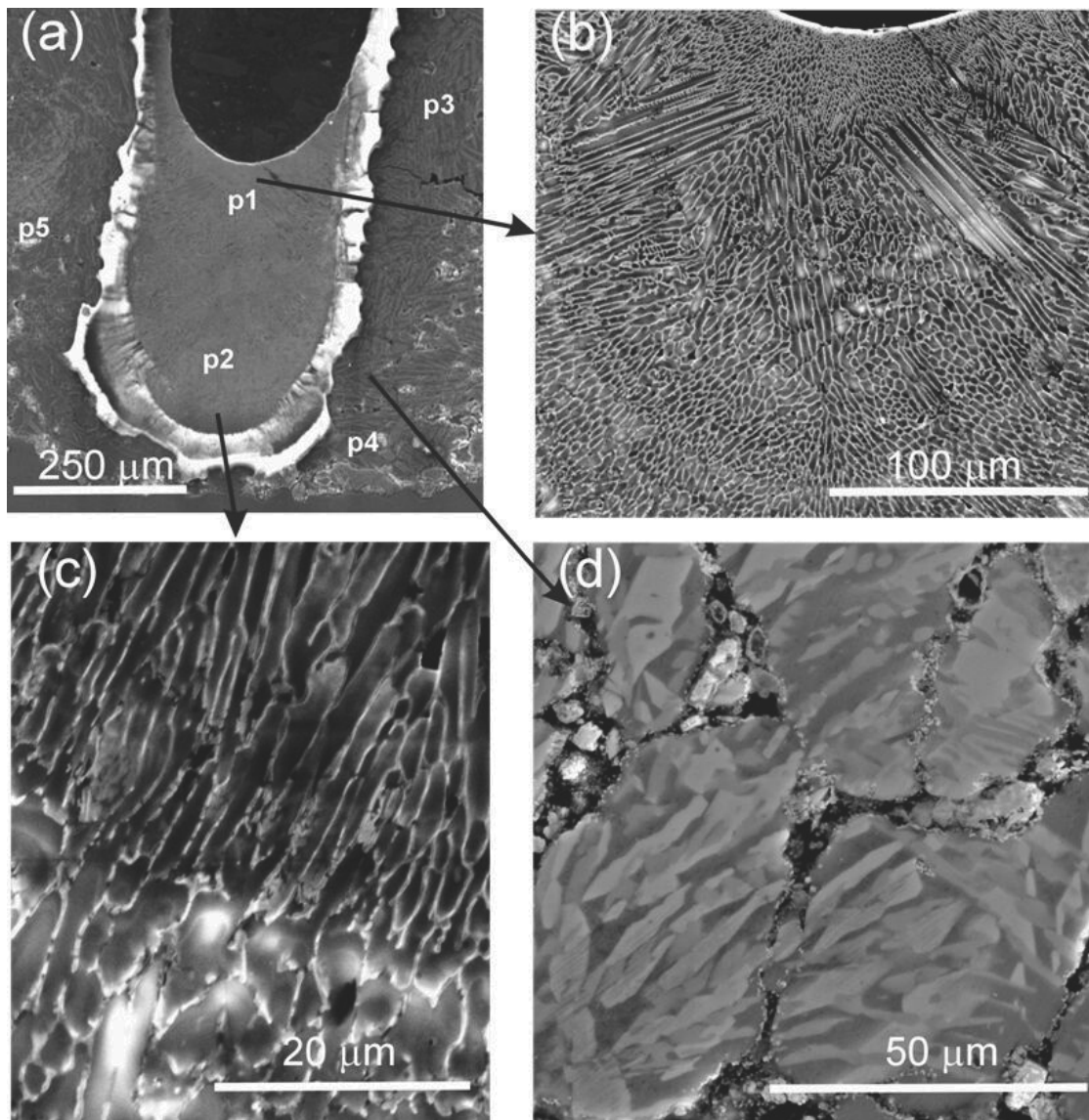
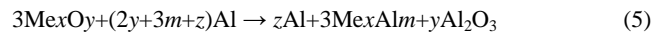


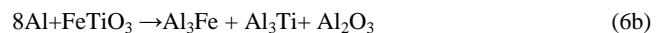
Fig. 6. An SEM micrographs of the microstructure of the overall cross-sectional laser alloying  $\text{AlFeTiO}_3$  composite powder incorporated in a aluminium matrix produced at  $10^5$  ( $\text{W}/\text{cm}^2$ )  $\text{CO}_2$  laser energy density and 6.5 cm/s laser beam speed

It can be found from these figures and from EDX microanalysis that in the middle part, (Fig. 6a,b), position p1 and p2, intermetallic compound  $\text{Al}_3\text{Ti}$  distribute in the  $\text{Al}_2\text{O}_3$  interdendritic region.

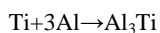
It was observed in Fig. 6a that due to the laser melting, the compact aluminium oxide zone migrates before the alloy crystallization front. Present results shows that around the remelted composite material behind the oxides' zone Fig. 6 (d), loosely joined grains of the composite powder occurred in which intermetallic phases were formed as a result of the heat coming from the self-propagating strong exothermic reaction [22,23]. The possible reactions of the oxides and aluminium can be described with a general formula:



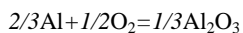
In which  $x, y, m$  is the molar number of the components. In our case, after laser remelting of the  $\text{Al-FeTiO}_3$  composite powder, intermetallic phases as well as their alloy and aluminium oxide are formed. Some examples of such reactions are as follows [16,24]:



and their accompanying free-energy changes:



$$\Delta G(\text{Jmol}^{-1}) = -52503 + 21,483 \cdot T$$



$$\Delta G(\text{Jmol}^{-1}) = -562,72 + 109,11 \cdot T$$

This indicates that the formation of  $\text{Al}_3\text{Ti}$  is more favorable than that  $\text{Al}_2\text{O}_3$ . The  $\text{Al}_3\text{Ti}$  is the first phase to be formed due to absorbed by composite powder the laser beam energy.

It can be seen that the exothermic reaction only exist directly in the laser radiation area. This indicates that the self-propagation reaction has been under the control of the laser processing parameters.

The best effects of laser remelting of the  $\text{AlFeTiO}_3$  composite powder were obtained when applying the laser energy flux per unit area  $I=5 \times 10^5 \text{ W/cm}^2$  and a scanning velocity of 6 cm/min.

## 4. Conclusions

Two different laser techniques have been presented here, with the use of which exothermic reaction were initiated by a laser beam in the  $\text{AlFeTiO}_3$  composite powder. Due to a different share of laser beam energy and the energy generated in the exothermic reaction, a different structure of the remelted  $\text{AlFeTiO}_3$  composite powder was obtained in those methods. In the first method, metaloceramic layers were obtained on the  $\text{AlSi}$ -alloy surface with a relatively good layer/substrate connection. However, cracks are formed on the connection boundary. This is connected with different coefficients of thermal expansion of the created metaloceramic layer on the metal substrate.

In the case of the other method and high intensity of the laser beam, complete melting of the  $\text{AlFeTiO}_3$  composite powder occurred in a large part. The melted material is uniform, without any pores. However, a partial segregation of chemical elements took place and on the boundary of the melted area, a layer of  $\text{Al}_2\text{O}_3$  oxides occurred, which constitutes a brittle connection with the metal substrate. This layer also reduces the flow of thermal energy. In consequence, unmelted powder grain is present on the boundary of the area completely remelted by energy generated as a result of exothermic reactions initiated with a laser beam and directly, by the laser energy.

The experimental results show that the effect of a continuous high intensity laser beam action on the  $\text{AlFeTiO}_3$  powder preplaced onto aluminium substrates is a multi-phase melting process. Because of very high degree of supercooling quick crystallization completed with the formation of a eutectic  $\text{Al-Fe-Ti-O}$  alloy.

It was found that advantageous microstructure of the remelted composite powder is obtained after its repeated remelting with a laser beam intensities higher than  $10^5 \text{ (W/cm}^2\text{)}$ . Successive scanning of a laser beam, the latter being treated in this case as a superficial source of energy moving at a constant velocity, induces a cycle of thermodynamic processes in the  $\text{AlFeTiO}_3$  powder and in the alloy.

The effects of this stage of the research project allow the presumption that the solution consisting in the application of a laser beam to remelt reactive composite powders can be applied to produce metaloceramic coatings of a dispersive and eutectic structure on aluminium substrate.

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