Synthesis of intermetallic compounds in the surface layer of eutectic silumin by dense plasma impact

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Particles reinforced metal matrix composites are one of the prospective groups of novel materials and they are widely used nowadays. Aluminium alloy matrix composites attract considerable attention due to their low density and low cost. At present transition metals trialuminides like Al_3Ti are considered as reinforcing particles in aluminium composites. Al_3Ti has a relatively high melting point (~1350 C), a low density (~3.3 g/cm^3) and a low coarsening rate at elevated temperatures. Thus, aluminium matrix composites reinforced by Al_3Ti particles could be used in conditions of more intense mechanic and thermal loads.

A large number of investigations were carried out in the area of synthesis of bulk aluminium matrix composites reinforced by metals trialuminides. At the same time many applications require good exploitation properties only of the materials surface layer. In this case techniques providing synthesis of composites in the surface layer are of interest. Treatment of a coating/substrate system by high energy particles ($\geq 10^6$ W/cm^2) is one of such techniques that allow to alloy the surface layer of a substrate with coating elements. Such treatment provides enough energy to melt a coating and the surface layer of a substrate. Convection in the melt leads to mixing and homogenization of elemental composition in the melted layer. A high cooling rate results in the formation of a disperse structure having submicron or nanocrystalline grains and phases. The change of the coating thickness and energy absorbed by the surface layer allows to control the concentration of coating elements in the mixed layer thus giving an opportunity to regulate phase composition and volume fraction of the synthesized phases. This approach was used to form the surface composite layer reinforced by Al_3Ti intermetallic particles in the Al-Si piston alloy and was realized by treating Ti coated alloy samples with compression plasma flows (CPF).

The samples used were made of a eutectic silumin alloy (12,9 Si; 3 Mg; 0,7 Cu; 0,4 Ni; 0,1 Fe; at.%, Al - balance). The titanium coating was formed using cathodic arc vapour deposition with the following process parameters: arc current of 100 A, bias voltage of -120 V, deposition time 10 and 20 min (the corresponding coating thickness 2.5 and 5.5 μm).

CPF were obtained using a gas-discharge magneto-plasma compressor of compact geometry. CPF treatment was performed in a ‘‘residual gas’’ mode in which the vacuum chamber was filled with nitrogen up to the preset pressure of 400 Pa. The plasma flow parameters were as follows: pulse duration ~100 μs, flow velocity $(5\div6)\cdot10^6$ cm/s and electron concentration $(4\div7)\ 10^{17}$ cm^{-3}. The discharge duration in the MPC amounted to ~ 100 μs. Treatment of the formed Ti/silumin system was carried out by three pulses. The energy density (Q) absorbed by the surface layer varied in the range of 4-19 J/cm^2 per pulse.

Element composition was analyzed by means of the Rutherford backscattering analysis of He^+ ions with the energy of 2 MeV. Phase composition of the samples was investigated by the X-ray diffraction analysis (XRD) in Bragg- Brentano geometry and Cu Kα radiation using a DRON 4-13 diffractometer. Surface and cross-section morphology as well as element composition were analyzed by means of scanning electron microscopy (SEM) using a LEO1455VP device equipped with an energy-dispersive X-ray Röntec detector. Microhardness

of the samples was tested by means of a Wilson Instruments 402MVD microhardmeter with a Vickers indenter under the load ranging from 0.1 to 1 N.

The data of the phase composition analysis showed that plasma impact with $Q \geq 7$ J/cm^2 led to melting in the Ti (2.5 μm) /silumin system, mixing of components and as a result – the formation of Al-Ti intermetallic phases (Fig. 1). The formation of $AlTi_3$ phase was found at Q=7 J/cm^2. The increase of Q up to 13 J/cm^2 resulted in additional formation of Al_3Ti compound with a tetragonal $D0_{22}$ structure. At Q=19 J/cm^2 the surface layer contained only Al_3Ti compound. Such a change of phase composition can be explained by the decrease of Ti concentration in the surface layer with the growth of Q. Treatment with Q=4 J/cm^2 did not lead to melting of the surface layer. At Q=7-13 J/cm^2 nonuniform mixing in the surface layer was observed by SEM. Local areas with a different concentration of titanium atoms were found thus resulting in the formation of different types of Al-Ti intermetallic phases during crystallization. The growth of the energy absorbed by the surface layer leads to the increase of the melted layer thickness and to the growth of the melt existence time. In case of a constant coating thickness the whole volume of the coating element will be distributed in the melted layer of a greater thickness resulting in the decrease of the alloying element concentration. That is consistent with the data of the elemental composition analysis. According to the energy-dispersive analysis Ti mean concentration in the layer with the thickness of ~1μm changes from 31±2 at.% at Q=13 J/cm^2 to 9±1 at.% at Q=19 J/cm^2. The increase of the melt existence time provides more uniform distribution of Ti atoms in the melt. Thus, at treatment parameters providing the highest energy absorbed by the surface layer only Al_3Ti was found.

The lattice parameters of Al_3Ti derived from the diffraction pattern are less than those of the standard (a=0.3853 nm and c=0,8583 nm). Q increase led to diminishing the Al_3Ti lattice parameters. At Q=19 J/cm^2 they were equal to: a=0,3792 nm and c=0.8488 nm. It is known that in Al_3Ti compound up to 15 % of Al atoms can be substituted by Si atoms thus leading to diminishing lattice parameters. It is seen from Fig. 1 that Q increase also resulted in diminishing Si diffraction lines intensity. Thus, some part of Si atoms can take part in precipitation from the melt of Al-Ti intermetallic phase which can be identified as $(Al,Si)_3Ti$.

Si atoms can also form an oversaturated solid solution on the aluminium basis crystallized in conditions of a high cooling rate thus resulting in the shift of Al diffraction lines to the region of greater diffraction angles (Fig. 1).

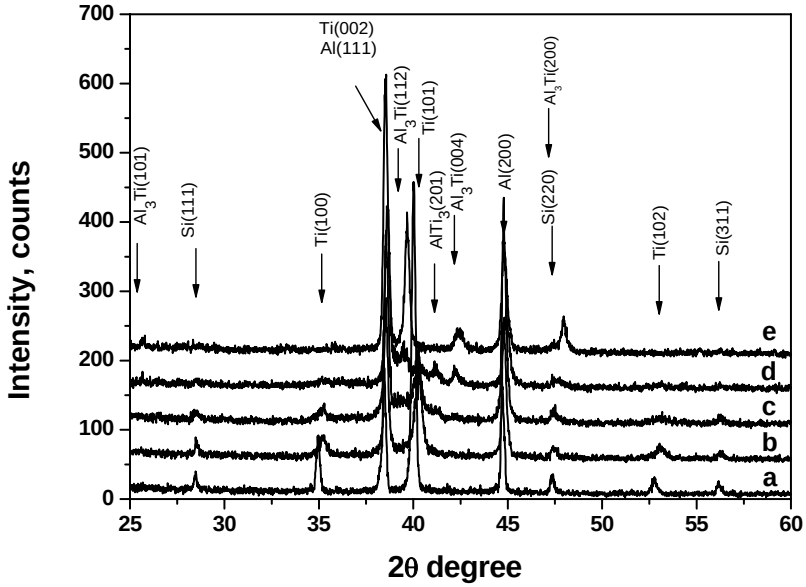

Fig. 1 XRD diffraction patterns of the Ti (2.5 μm) /silumin system samples before (a) and after CPF treatment with the energy density of 4 J/cm^2 (b), 7 J/cm^2 (c), 13 J/cm^2 (d), 19 J/cm^2 (e).

Similar dependencies were observed in the samples of the Ti (5.5 μm) /silumin system treated by CPF at Q=7-19 J/cm^2 but Al_3Ti or $(Al,Si)_3Ti$ was the only intermetallic phase observed (Fig. 2).

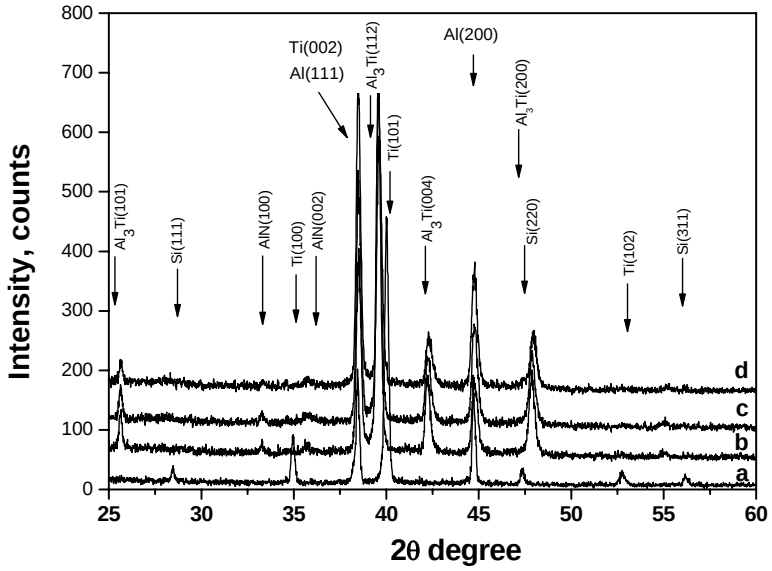

Fig. 2 XRD diffraction patterns of the Ti (5.5 μm) /silumin system samples before (a) and after CPF treatment with the energy density of 7 J/cm^2 (b), 13 J/cm^2 (c), 19 J/cm^2 (d).

The investigation of cross-section morphology showed that the thickness of the composite layer in the system Ti (5.5 μm) /silumin treated by CPF at Q=19 J/cm^2 was 50-60 μm (Fig. 3). The energy-dispersive analysis along the cross section showed homogeneous distribution of Ti and Si in the melted layer (assuming resolution limit ~ 1μm). One can see that $(Al,Si)_3Ti$ precipitates have relatively uniform distribution in the composite layer.

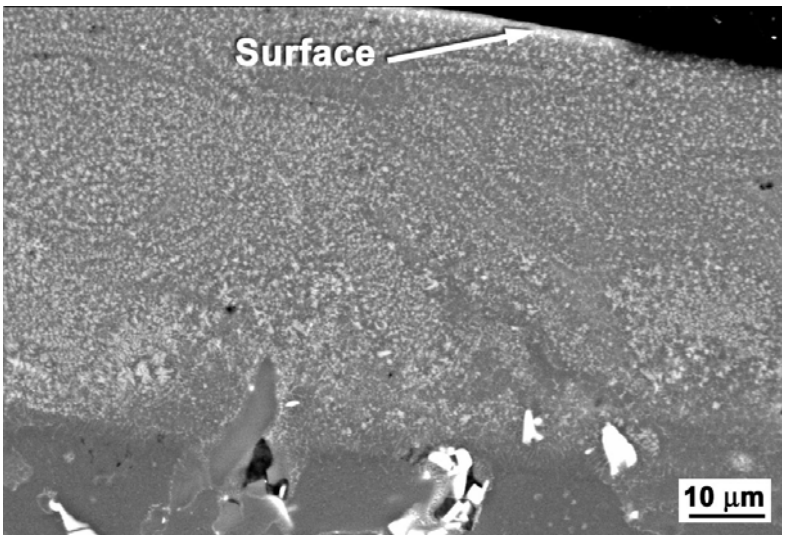

Fig. 3 Cross-section morphology of the Ti (2.5 μm) /silumin system sample after CPF treatment with the energy density of 19 J/cm^2 (e).

$(Al,Si)_3Ti$ precipitates grew in the form of dendrites due to high undercooling (Fig. 4a). Their size varied in the range of 0.2 – 1 micrometers depending on plasma parameters for the samples with 2.5 μm Ti coating. The increase of the Ti coating thickness up to 5.5 μm allowed to

form a surface layer consisting only of intermetallic phase dendrites with the size up to 4 μm after CPF treatment at Q=7 J/cm^2 (Fig. 4b).

a)

b)

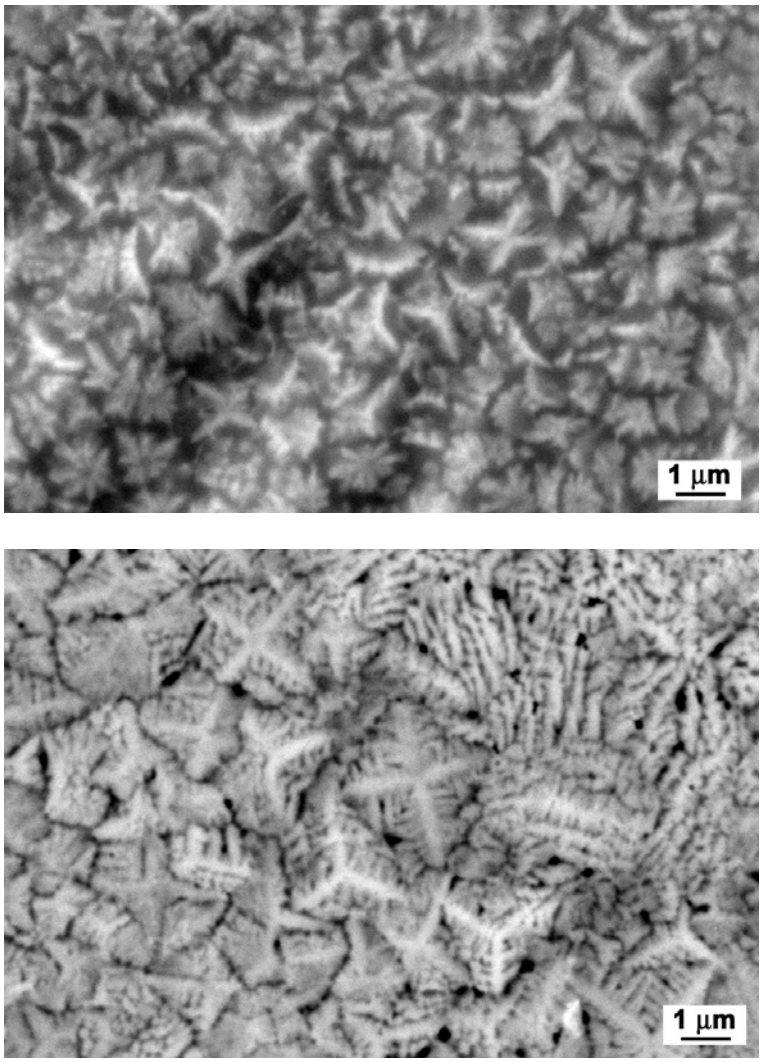

Fig. 4 Cross-section morphology of the Ti (2.5 μm) /silumin system (a) and Ti (5.5 μm) /silumin system samples after CPF treatment with the energy density of 19 J/cm^2 (a) and 7 J/cm^2 (b).

The microhardness tests showed that CPF treatment resulted in the surface microhardness increase from 1.3 GPa (for the initial silumin sample) to 2.2 GPa (for the Ti (2.5 μm) / silumin system sample treated at Q=19 J/cm^2) and to 4.4 GPa (for the Ti (5.5 μm) / silumin system sample treated at Q=7 J/cm^2). The latter value lies in the range attributed to microhardness of bulk Al_3Ti, which agrees with the data of SEM investigations (Fig. 4b).

The results of this work demonstrate that compression plasma flows treatment of Ti pre-coated silumin samples allows to form a surface composite layer with the thickness up to 60 μm reinforced by $(Al,Si)_3Ti$ intermetallic particles. The size of intermetallic precipitates (mainly dendritic-like) varies in the range of 0.2 – 4 micrometers depending on treatment parameters and Ti coating thickness. Plasma treatment also results in dissolution of primary silicon crystals and formation of a Al(Si) supersaturated substitutional solid solution. The change of treatment parameters and Ti coating thickness allows to control Ti atoms concentration and volume fraction of reinforced particles providing a substantial microhardness increase up to 4.4 GPa.