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Premixes production for synthesis of wear-resistant composite materials

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Abstract. State of the art line of powder metallurgy is application of initial powders as micro-composites with additional components - premixes. Usage of premixes inhibits segregation of added components and implies the homogeneity of powder charge composition, and finally it has a significant impact on structure formation and properties of end products. The aim of the present work was to design the new production technology of premixes based on iron powder which is layer-by-layer plated by aluminium and copper. We propose to carry out production of Cu-Al-Fe premixes in two stages: cladding of iron powder by aluminium and coating of the obtained composite by copper. The self-developed technique of vibration treatment of iron and aluminium powder mixture was chosen for this purpose. The uniform in thickness and unbroken copper-plating of Fe-Al powders were carried out by chemical technique. Physico-chemical properties and production conditions of premixes-powders were studied, besides optimal parameters of production and further heat-treatment were selected. In the result of the present study the Fe-Al-Cu premixes with laminated structure comprising of iron core, Fe-Al and Cu-Al intermetallide shells were synthesised.

1. Introduction

State-of-the-art problems are development of new multifunctional coatings with required complex of properties, e.g. improved corrosion, abrasive resistance and fatigue resistance, low friction factor, as well as designing of appropriate techniques and machines for production of such materials. From this point of view it appears that the most promising direction for production of composites is powder metallurgy, since its techniques have almost an unlimited number of choices for composition and materials structure. The state of the art line of powder metallurgy is application of input powders as micro-composites with additional components - premixes. Usage of premixes, what inhibits of added components segregation and implies the homogeneity of powder charge composition, has a significant impact on structure formation and properties of end product. For example, the strength and ductility of the steels made of premixes comprised of iron powder plated by Ni-Cu alloys nanocrystals are in 2-4 times higher than steels with the same composition, but which were produced of powder mixture.

Typically, wear-resistant materials have composite structure comprising of plastic matrix and solid inclusions, for instance, copper and intermetallides, respectively. Wear-resistant composites based on copper with inclusion of reinforcing premixes in the form of Cu-C [2], Al-Cu-Fe quasicrystals doped by B and Al_2O_3 [3] granulates and other compositions are generally known.

The aim of the present work was to design the new technology of premixes production based on iron powder which is layer-by-layer plated by aluminium and copper. It was assumed that composites with copper matrix and produced inclusions will have improved mechanical properties owing to formation of Cu-Al premixes and Fe-Al intermetallides in the result of Cu-Al-Fe sintering.

2. Experimental

Aluminium powders PA-4 and PAD-6, PGR 3.160.28 iron and PMS-1 copper powders were used as input materials. Production of Cu-Al-Fe premixes was carried out in two stages: cladding of iron powder by aluminium and coating of the obtained composite by copper. Coppering of Fe-Al premixes was carried out at room temperature by contact technique, namely, copper ions were substituted by iron ones in solution of CuSO4 sulfuric acid which were acidified by 0.3% H2SO4.

Plating of iron powder by copper was performed by the self-developed technique of vibration treatment of iron and aluminium powder mixture [4]. This technique allows us to vary process parameters in the wide range and consequently it provides to obtain predetermined structures and phase composition of cladded powders.

Appearance of apparatus for mechanical plating of powder is shown in Figure 1. The main parts of apparatus are vibrator with crank drive and DC motor, which allow regulating frequency and amplitude of vibration to be made smoothly. Rotating velocity of motor determines the vibration frequency; the value of crank eccentricity is responsible for amplitude. Airtight cylinder of stainless steel with pipes providing the predetermined atmosphere is placed on vibrator. Inductive unit with transformer type VCIN-15 is used for heat treatment of powders after hitting.

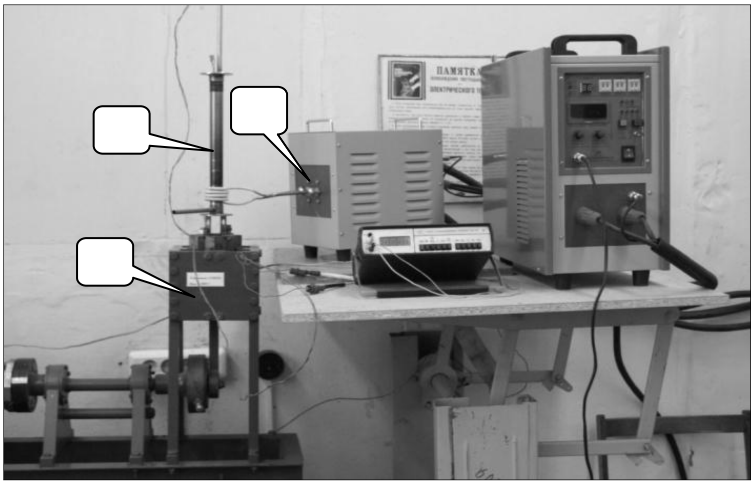

Figure 1. Appearance of apparatus for mechanical cladding of powders: 1 - vibrator unit, 2 - airtight cylinder for powders (plating unit), 3 - inductive heater VCIN-15

In essence the apparatus induces vibratory movements with high amplitude up to 10 mm and operates within the frequency range 30-50 Hz. Reciprocating motion imparts to powder mixture, but the frequencies of plating unit, iron and aluminium powders are different. Whereas the lifting height of the powder particles is up to 200-250 mm. Particles of different composition have various velocities and vibration phases, they have both tangent and central collisions. At the tangent collisions in the result of the tangential stresses between particles the rubbing of softener aluminium on iron powder particles occurs, when in the result of the central collisions the local plastic deformation is taking place, the latter one provides the intensification of diffusion processes [5].

The following instruments and techniques were applied to make our experiments. The frequency of plating unit vibration was determined through rotating velocity of motor which was measured by contactless tachometer CEM-AT-8. Heating temperature of powder mass was determined as the temperature of plating unit walls, and it was measured by Kelvin IR pyrometer with inaccuracy of ±1%. The surface of plated powder was observed on MEIJI EMZ-13TR stereomicroscope. The microstructure observation were conducted using Olympus GX-51optical microscope equipped with the SIAMS system of image analysis, the phase composition analysis of premixes were undertaken using CARL ZEISS EVO 40 scanning electron microscope. The velocity of collided particles was calculated according to the technique described in the literature [6]. The contact time of collided spheres (τ), the closest approach at the impacts (a_m) and the values of plastic and elastic deformations (p_m) were calculated in accordance with equations (1, 2, 3) [7]:

$$\tau = 4{,}53\left[\frac{(\delta_1+\delta_2)m_1m_2}{(m_1+m_2)}\right]^{\frac{2}{5}}\left(\frac{R_1+R_2}{V_0R_1R_2}\right)^{\frac{1}{5}} \quad (1)$$

where $\delta \equiv \frac{1-\mu^2}{E\pi}$; μ is Poisson's ratio; E is Young's modulus; m_1 and m_2 are the masses of collided spheres; R_1 and R_2 are the sphere radii; v_0 is the spheres impact velocity,

$$a_m = \left[\frac{15\pi v_0^2(\delta_1+\delta_2)m_1m_2}{16(m_1+m_2)}\right]^{\frac{2}{5}}\left(\frac{R_1+R_2}{R_1R_2}\right)^{\frac{1}{5}} \quad (2)$$

$$p_m = 0{,}2515\left[\frac{v_0^2}{(\delta_1+\delta_2)^4}\cdot\frac{m_1m_2}{(m_1+m_2)}\cdot\left(\frac{R_1+R_2}{R_1R_2}\right)^3\right]^{\frac{1}{5}} \quad (3)$$

To calculate depth and degree of plastic deformation of every sphere we used the mentioned above data in the process of iteration, besides the growth of the yield strength was taken into account at a given deformation rate.

3. Results and discussion

3.1. Selection of optimal characteristics of plating unit vibration and treatment time

Experiments were carried out within the ranges of vibration frequency and amplitude 20-50 Hz and 2-10 mm, respectively. The airtight quartz tube of the same diameter as plating unit was used to monitor the behaviour of powders. Vibro-boiling process with the separation effect was caused at all applied frequencies when the variation of amplitude was within the range from 2 to 6 mm. It is interesting to note that more dense iron powder was raised, but lesser dense aluminium particles were sunk. When amplitude is higher than 6 mm the vibro-boiling is substituted by vibration motion of powder particles, whereas the increase of vibration frequency is accompanied by the particles lifting height rise. The lifting height is up to 200-250 mm at the amplitude of airtight cylinder about 8-10 mm and frequency about 35-40 Hz, besides at these parameters the vibration phases of powders with diverse densities are changed. The most efficient plating process is performed at such vibration characteristics of airtight cylinder. Further increase of frequency was not tested due to high dynamic loads on part of the apparatus.

The optimal time of treatment in plating unit is 240-300 seconds. During shorter period of treatment some particles remain free from coating, and no positive effect was observed in the result of longer period of particles processing

3.2. Influence of ratio of dispersion and volume of interacted powders on plating efficiency

For plating experiments powders with different particles sizes were used: iron powders – 140÷160 μm, 100÷140 μm, 70÷100 μm, 40÷70 μm; aluminium powder: 140÷160 μm, 100÷140 μm, 70÷100 μm, 40÷70 μm and 1÷10 μm.

The cladding is nonuniform and broken when for treatment iron and aluminium powders had close particles sizes; such characteristic view is shown in figure 2.

The most uniform and unbroken plating of iron powder was obtained at the particles sizes of iron and aluminium within the ranges 100÷140 and 1÷10 μm, respectively.

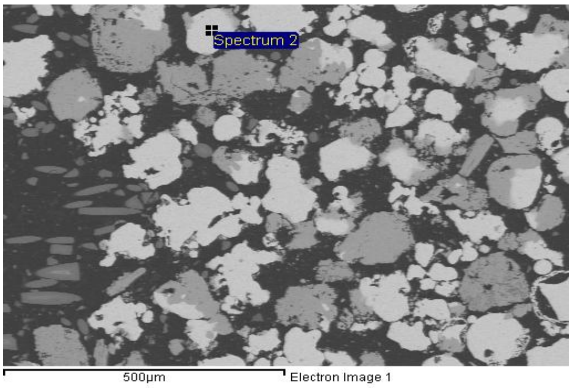

Figure 2. Iron powder with aluminium shell, where fractions of Fe and Al powders were 100÷140 µm and 70÷100 µm, respectively. Aluminium is dark grey.

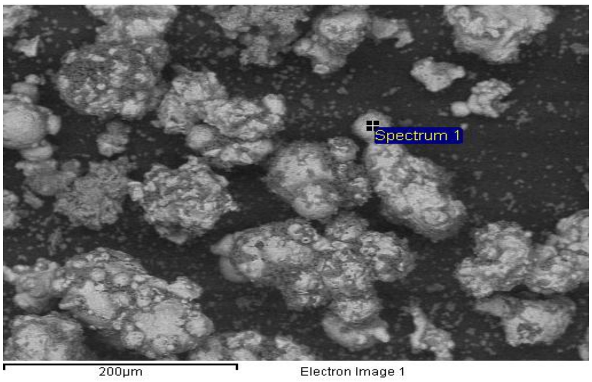

Figure 3. Iron powder plated by aluminium one, where fractions of Fe and Al powders were 100 – 140 µm and 1÷10 µm, respectively. Aluminium is dark grey.

It is the author's opinion that in this case the iron powder play role of impact tool which provides rubbing and hammering of aluminium powder particles placed between two collided iron particles.

In the result of the present investigation of influence of quantitative volumetric powder ratio on quality and efficiency of plating it was established that volumetric ratio, when powders of 40÷160 µm are used, has no significant effect on cladding process. However, to avoid additional operation of cladded powder separation of excess aluminium it is necessary to maintain powder volumetric ratio Al/Fe=1/4÷1/5.

The effect of 1÷10 µm highly-dispersed aluminium powder is significantly different. When the Al/Fe ratio is higher than 1/4 the cladding process stops, since aluminium powder is tamped down the plating unit and iron powder particles are sunk in it. For this reason the optimal ratio of volumes is Al/Fe=1/5÷1/6 when highly dispersed aluminium powder is used.

3.3. Diffusive interaction of powders

Iron and aluminium powders were sharply heated up in plating unit to provide solid-phase interaction of particles. Earlier it was established [5] that high-rate heating, which is higher than 50 ^{o}C per second, leads to increase of real diffusivity. Hence the rate of Fe-Al solid-phase reaction is increased and the treatment time becomes shorter, respectively. Thermal treatment on air leads to iron oxidation; hence the plated powders were heated in hydrogen and nitrogen atmospheres which delay the reactive diffusion. Several parameters were varied in the experiment, namely, the top temperature 650-850^{o}C, the exposure time 30-40 s, heating rates $^{o}Cs^{-1}$. In the result of the experiments it was established that efficiency of solid-phase reactions in hydrogen and nitrogen is almost equal, thus further experiments were carried out in a nitrogen atmosphere.

Characteristic view of iron particles cladded by aluminium after 300 s mechanical treatment, and subsequent 10 s thermal treatment at 700-725^{o}C is shown in Figure 4.

Further increase of both temperature and exposure time leads to rise of diffusion rate and multiphase Fe-Al alloys are obtained, example of this is shown on Figure 5.

The view of ferroaluminide powder cladded by copper and its phase composition are submitted in Figures 6 and 7, respectively.

Copper-plated ferroaluminium powders were thermally treated to provide copper interaction with ferroaluminium core. Particles phase composition of this powder after heat treatment at 700°C during 15 minutes is shown in figure 8.

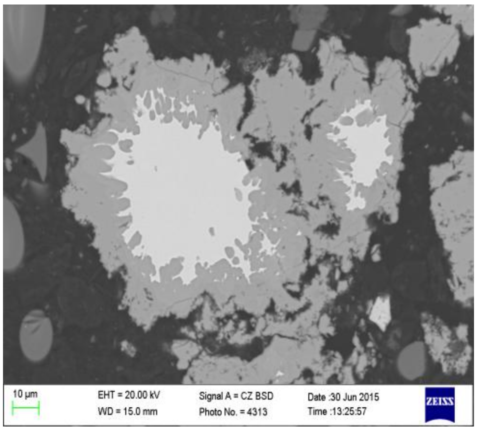

Figure 4. Iron particles plated by aluminium after solid-phase reaction, where dark gray is Fe_2Al_5 intermetallide and light gray core -Fe.

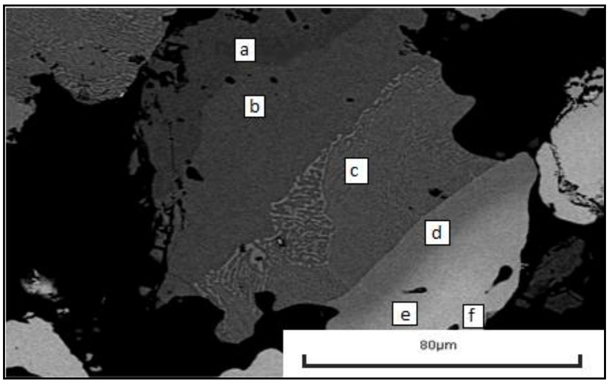

Figure 5. Iron particles plated by aluminium after 40 s heating at the temperature 850°C with following composition (at. %): a - ferroaluminide (FA) of Al 69 and the balance Fe , b - FA of Al 61 and the balance Fe , c - FA of Al 51 and the balance Fe, d - pure iron.

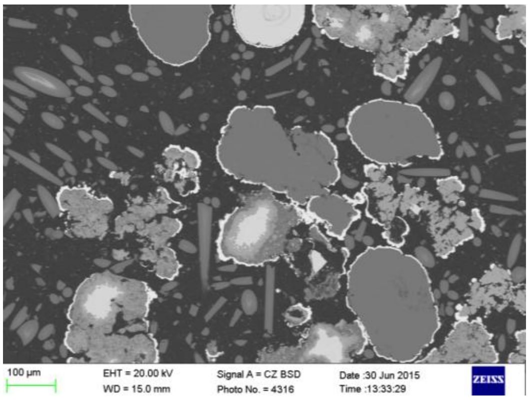

Figure 6. The Cu-Al-Fe composite powder

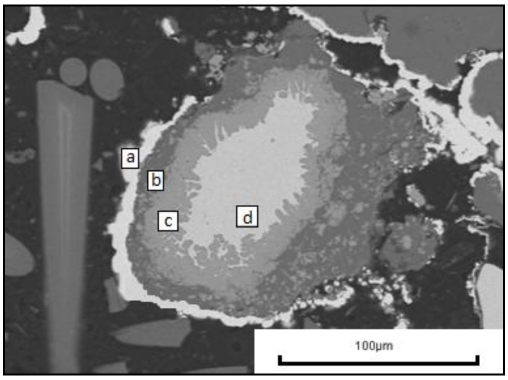

Figure 7. Phase composition (at.%) of FA powders plated by copper: a - Cu 99 and the balance Fe ; b - Al 99 and the balance Fe ; c - Al 75 and the balance Fe ; d - pure iron.

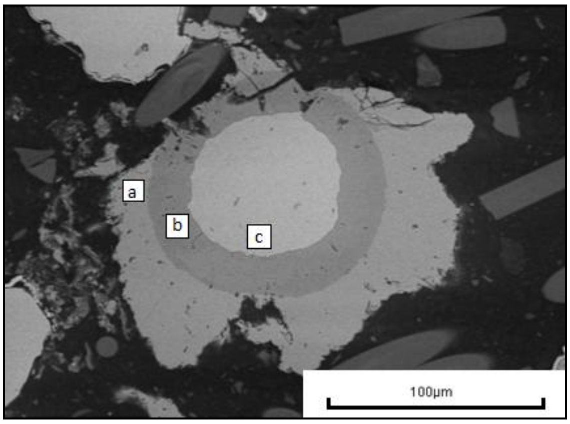

Figure 8. Phase composition (at.%) of the Cu-Al-Fe premix after final thermal treatment: a - Cu 25, Al 62 , Fe 13; b - Cu 4, Al 69 %, Fe 27; c - pure iron

4. Conclusions

High-performance vibration technique was proposed and mechanical parameters of iron powder plating by aluminium were selected. In the result of experiments the powder dispersion parameters and powder volumetric ratios were determined for efficient cladding. A number of conditions of heat treatment, namely, heating rate, top temperature and exposure time, were optimized to obtain in plating unit the Fe-Al intermetallic compounds of predetermined composition. The uniform in thickness and unbroken copper-plating of Fe-Al powders were carried out by chemical technique. Diffusion of iron and aluminium into copper shell from the Fe-Al core were revealed after heat treatment of the Fe-Al-Cu powders obtained chemically. Premixes of Fe-Al-Cu were obtained with laminated structure comprising of iron core, Fe-Al and Cu-Al intermetallide shells of variable composition.

Acknowledgements

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