INFLUENCE OF SURFACTANTS ON PHASE FORMATION DURING PRODUCTION OF Al–Cu–Fe SYSTEM POWDERS FOR THERMAL COATINGS BY THE METHOD OF MECHANOCHEMICAL SYNTHESIS

A.N. BURLACHENKO and **Yu.S. BORISOV**
E.O. Paton Electric Welding Institute, NASU, Kiev, Ukraine

Influence of the quantity and methods of adding surfactants on phase formation during mechanochemical synthesis of composite powder of $Al_{70}Cu_{20}Fe_{10}$ system was studied. It is established that surfactant application allows avoiding sticking of charge compounds to the drum wall and intensifies the process of new phase formation.

Keywords: *thermal coatings, powders, mechanochemical synthesis, surfactants, sticking, phase composition, quasicrystalline phase*

One of the urgent trends in thermal spraying (TS) is development of the technology of deposition of coatings with special structure types (nanocrystalline, nanocomposite, quasicrystalline) [1–4]. One of the stages of development of such technologies is application of powders of a new type ensuring under TS conditions formation of coatings with specified structure type. Among the methods of producing such powders, widely applied are those of mechanical alloying and mechanochemical synthesis (MCS), which are based on phenomena of repeated processes of cold welding and crushing of particles of obtained material components during their processing in high energy ball mills (attritor, planetary mill). Examples of mechanical alloying application are producing powders, for instance, of FeCr–TiCN system to form nanocomposite coatings [5], and of MCS application — producing powders, for instance, of Al–Cu–Fe system to form coatings of a quasicrystalline structure [5–7]. However, in case of processing during MCS a mixture of powders of Al–Cu–Fe system, containing ductile metals (aluminium, copper, iron), a phenomenon of charge particle sticking to the drum wall and planetary mill crushers is observed. As a result, part of the powder is eliminated from MCS process, thus lowering both process efficiency and its effectiveness. To eliminate the phenomenon of sticking and intensify phase formation process, additives of surfactants (SA) to the powder mixture are used. Here, SA amount and method of their addition to the charge should not only prevent charge sticking to the drum wall and crushers, but also ensure the equilibrium of the multiple process of welding and crushing of the formed composite conglomerate, which exactly results in new phase formation.

This work gives the results of investigation of SA influence on phase composition and structure of par-

Table 1. SA characteristics

SA	T_m, °C	T_b, °C	σ, mN/m	γ, g/cm^3
Oleic acid $CH_3(CH_2)_7CH = CH(CH_2)_7COOH$	13.40	228.0	32.8	0.895
Ethyl alcohol C_2H_5OH	−114.65	78.3	22.8	0.789

Table 2. Modes of SA addition during MCS (5 h duration)

No.	SA	Method of SA addition	SA amount, wt.%	Product phase composition
1	–	–	–	AP, β, θ, ω, ψ
2	Oleic acid	Once	1	Same
3		Same	5	23 % AP + 77 % (44 % ψ-phase + 56 % β-phase)
4		Periodically, by 1 wt.% every hour of processing	5	27 % AP + 73 % (68 % ψ-phase + 32 % β-phase)
5	Ethyl alcohol	Once	1	AP, β, θ, ψ, ω
6		Same	5	18 % AP + 82 % (41 % ψ-phase + 59 % β-phase)
7		Periodically, by 1 wt.% every hour of processing	5	22 % AP + 78 % (60 % ψ-phase + 40 % β-phase)

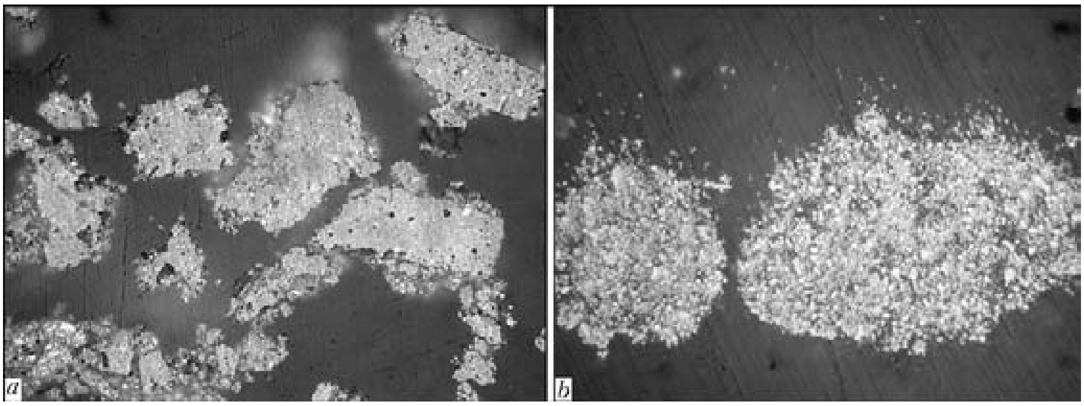

Figure 1. Microstructures (×1000) of powder particles of Al–Cu–Fe system produced in MCS processes without (*a*) and with (*b*) SA (oleic acid; periodical addition by 2 wt.% every hour of treatment)

ticles during MCS of powder mixture of Al–Cu–Fe system, close by its composition to the region of existence of quasicrystalline ψ-phase.

Experimental procedure. To study SA influence on phase formation at MCS of powders of Al–Cu–Fe system, corresponding by their composition to $Al_{70}Cu_{20}Fe_{10}$ (at addition of extra iron as a result of crushing, it approaches $Al_{63}Cu_{25}Fe_{12}$ of a quasicrystalline structure), initial powders of PA-4 aluminium (40–60 μm), PMS-1 copper (20–40 μm) and PZhR iron (100–160 μm) were used. MCS process was conducted in Activator 2-SL planetary mill at drum rotation speed v_{dr} = 1500 rpm, and ratio of spheres weight to charge weight of 10:1 for 5 h. Used as SA were oleic acid and ethyl alcohol (Table 1) in the amount from 1 up to 5 wt.% with different methods of SA addition (Table 2).

MCS process was periodically interrupted (every hour of processing), processed charge was removed, and in case of its sticking to the drum wall, it was

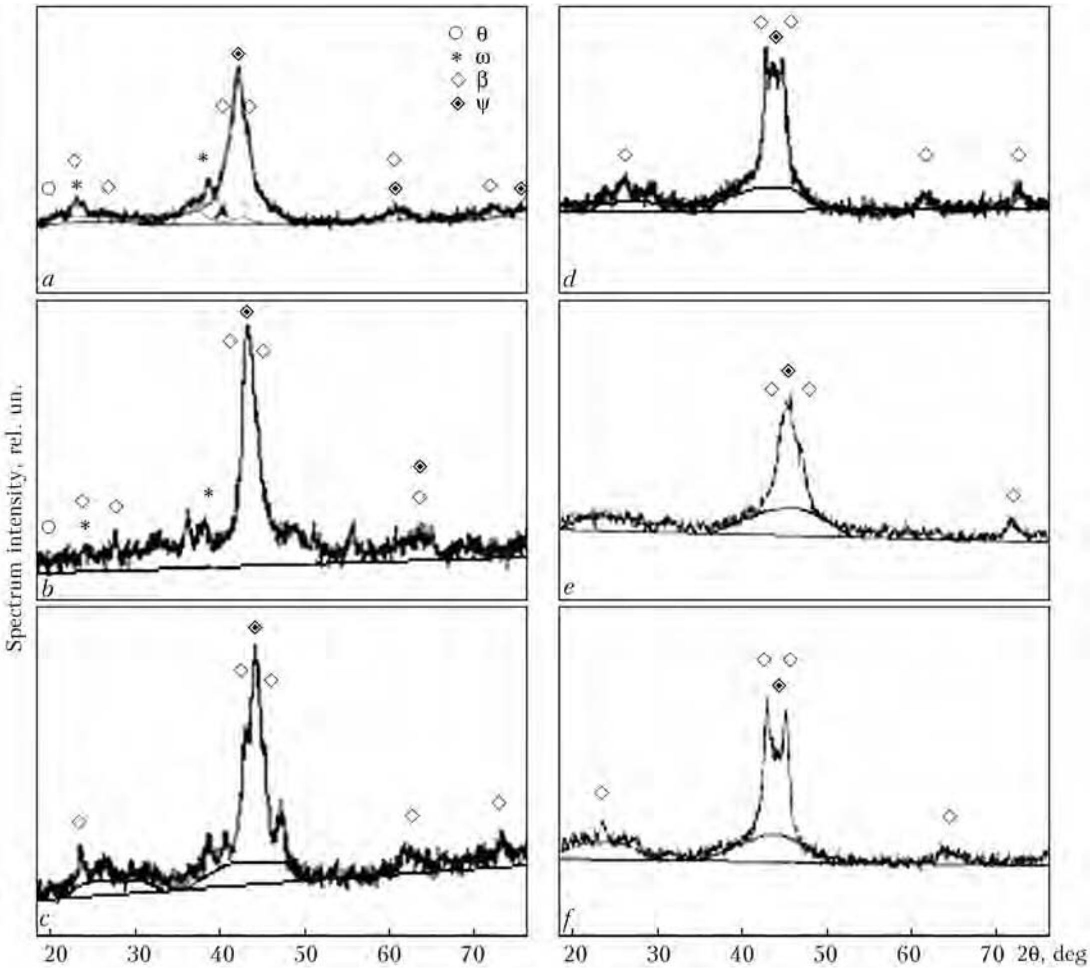

Figure 2. Roentgenograms of powders of Al–Cu–Fe system obtained by MCS at v_{dr} = 1500 rpm for 5 h without SA (*a*) and at different SA addition methods: once by 1 (*b*) and 5 (*c*, *e*) wt.%; periodically by 1 wt.% every hour of MCS (*d*, *f*) with oleic acid (*b*, *e*, *f*) and ethyl alcohol (*c*, *d*)

knocked off and crushed in a ceramic mortar, poured into the drum, and MCS process was carried on. Temperature measurement inside the drum was conducted with UT-70V multimeter. Synthesized powders were studied using metallography (Neophot-32 optical microscope with digital photography attachment) and X-ray structural phase analysis (DRON-UM1 diffractometer with monochromatic CuK_α-radiation).

Experimental results. Temperature measurement inside the drum directly after MCS showed that in all the modes of SA addition, it practically did not change, and was equal to 70–75 °C.

During treatment of initial powders without SA application, continuous sticking of up to 5–10 mm layer to drum wall is observed. Here, a multiphase system forms in the end product (Table 2, No.1; Figure 1, *a*; Figure 2, *a*).

After operation for 1 h with addition of SA in the amount of 1 wt.%, no sticking is observed, particles are of a round shape with average size of about 1 mm. Further continuation of the process leads to sticking, and, as a consequence, to producing the end product similar to the one produced without SA application (Table 2, Nos. 2, 5; Figure 2, *b*).

Periodical addition of 1 wt.% of SA every hour of MCS in the total amount of 5 wt.% (Table 2, Nos. 4, 7) leads to absence of sticking during the entire synthesis process, powder particles have a shape close to the round one with finely-dispersed structure (Figure 1, *b*) and phase composition, consisting of amorphous (AP), quasicrystalline ψ- and crystalline β-phases (Table 2, Nos. 4, 7; Figure 2, *d*, *f*).

One-time addition of 5 wt.% of SA (Table 2, Nos. 3, 6) leads to absence of sticking in the first 3 h of mechanical processing, the powder being loose, and in some places small local heating of MCS products up to 200–250 °C occurs at charge unloading from the drum. MCS continuation leads to sticking of a thin layer of about 2–3 mm to the drum wall after 1 h, and after 2 h — to sticking of a layer of up to 5–10 mm thickness to the drum wall. Final product of synthesis, similar to periodical addition of SA, consists of AP, quasicrystalline ψ- and crystalline β-phases (Table 2, Nos. 3, 6; Figure 2, *c*, *e*).

Thus, it is established that SA application in production of Al–Cu–Fe system powders for TS by MCS method promotes an intensification of the process of new phase synthesis and formation of spherical particles of 20–40 μm size. An optimum mode of SA addition, preventing sticking of processed material during the entire time of MCS (5 h) at processing of a mixture of Al–Cu–Fe system, is a regular periodical (every hour) addition of SA in the amount of 1 wt.%. In this case, maximum content of quasicrystalline ψ-phase of 68 wt.% in MCS product was achieved. No essential difference between the influence of oleic acid and ethyl alcohol on the process of new phase synthesis at MCS in a mixture of Al–Cu–Fe system was found.

1. Tjong, S.C., Chen, H. (2004) Nanocrystalline materials and coatings. *Materials Sci. and Eng.*, **1/2**, 1–88.
2. Ragulya, A.V., Skorokhod, V.V. (2007) *Consolidated nanostructured materials*. Kiev: Naukova Dumka.
3. Moya, J.S., Lopez-Esteban, S., Pecharroman, C. (2007) The challenge of ceramic-metal microcomposites and nanocomposites. *Progress in Material Sci.*, **52**, 1017–1090.
4. (2005) Thermal spray coatings having quasi-crystalline phase, properties and applications (Review). *Fizyka i Khimiya Tv. Tila*, 6(**1**), 124–136.
5. Borisov, Yu.S., Borisova, A.L., Adeeva, L.I. et al. (2010) Producing of powders for thermally sprayed coatings by methods of mechanical alloying and nanomechanical synthesis. *Svarochn. Proizvodstvo*, **12**, 18–22.
6. Borisova, A.L., Borisov, Yu.S., Adeeva, L.I. et al. (2008) Effect of parameters of mechanochemical synthesis on structure, phase composition and properties of thermal spraying of Al–Cu–Fe system powders containing quasicrystalline phase. *The Paton Welding J.*, **9**, 14–21.
7. Borisova, A.L., Adeeva, L.I., Tunik, A.Yu. et al. (2009) Investigation of thermal spraying of Al–Cu–Fe–Ti–Cr–Si system powders produced by mechanochemical synthesis with subsequent annealing. *Poroshk. Metallurgiya*, **9/10**, 31–40.