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FORMATION OF COATINGS OF THE FeTi–SiC SYSTEM DURING THERMAL SPRAYING OF POWDER PRODUCED BY THE METHOD OF MECHANOCHEMICAL SYNTHESIS

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ABSTRACT

The study of the formation of composite coatings of the FeTi–SiC system, produced by spraying using thermal spraying methods: subsonic (plasma) and supersonic (detonation), was carried out. Composite powder, produced by the method of mechanochemical synthesis of ferrotitanium and silicon carbide powder mixture, was used as the feedstock for spraying. Selection of the composition of powder mixture of ferrotitanium and silicon carbide for the process of mechanochemical synthesis was carried out on the basis of thermodynamic calculations of changes in the values of isobaric-isothermal potentials (Gibbs energy) of reactions in the FeTi–SiC system with selection of the reaction whose passage is the most favorable from a thermodynamic point of view. As a result of mechanochemical synthesis, a composite powder of the FeTi–(SiC, TiC, Ti₂Si₃) system with an amorphous-nanocrystalline structure was produced. Heterogeneous composite coatings consisting of a Fe₂Ti metal matrix with uniformly distributed ceramic inclusions of titanium carbide and silicide and oxide components were produced by plasma and detonation spraying methods. By studying the mechanical characteristics, it was established that the produced thermal sprayed coatings of the FeTi–SiC system are classified as materials with a microcrystalline heterophase structure by the type of structural state. The microhardness of the coatings is 8.5 and 8.0 GPa for plasma and detonation coatings, respectively.

KEYWORDS: mechanochemical synthesis, thermal spraying, composite powder, coating, mechanical properties

INTRODUCTION

Degradation of the surface of parts caused by the influence of aggressive media (abrasive wear, corrosion, high temperature) leads to a reduction in the service life of equipment as a result of the failure of its components [1, 2]. Increasing the service life of equipment can be achieved by creating a protective layer on the surface of parts that is resistant to mechanical, chemical and thermal effects. The combination of toughness and plasticity of the metal matrix with hardness, corrosion, wear and heat resistance of ceramic inclusions makes metal-ceramic (cermet) materials promising for use as a protective layer and allows expanding the service characteristics of parts [3]. At present, iron and its alloys, as well as ferroalloys, are widely used as a metal matrix, which ensure lower production costs for metal-ceramic materials [4–9]. High values of hardness and Young's modulus, good resistance to corrosion and oxidation make silicon carbide a promising candidate for use as a ceramic component in metal-ceramic materials [10].

Thermal spraying (TS) technology is an effective and economical method to produce a metal-ceramic protective layer on the surface of a part [11]. The main difficulties in the formation of metal-ceramic coatings are weak interfacial bond between ceramic inclusions

and a metal matrix, uneven distribution of dispersed ceramic inclusions in the matrix, and also the risk of interfacial interaction, which leads to the formation of undesirable compounds and coating embrittlement [12]. The use of mechanochemical synthesis (MChS) technology allows producing composite powders (CP), which can be used in TS of protective coatings with a homogeneous structure [13].

The aim of the work was to study the process of coating formation under thermal spraying methods using a powder of the FeTi–SiC system produced by mechanochemical synthesis and to determine the mechanical properties of the resulting coatings.

MATERIALS AND RESEARCH PROCEDURE

FeTi–SiC composite powder, produced by the MChS method from ferrotitanium powders (Ti content — 47 wt.%; particle size — 63–100 μ m) and silicon carbide (particle size ~3 μ m)was used as the feedstock for thermal spraying, (Figure 1). The microhardness of FeTi particles is in average 3.8 ± 1 GPa, and that of SiC particles is 30 GPa.

The phase analysis of the initial powders showed (Figure 2) that the silicon carbide powder has a single-phase structure, and ferrotitanium has a two-phase structure consisting of FeTi and Fe_0 , $Ti_{0.8}$ phases.

The MChS process was carried out in a planetary mill (Activator-2S1) at a drum rotation speed

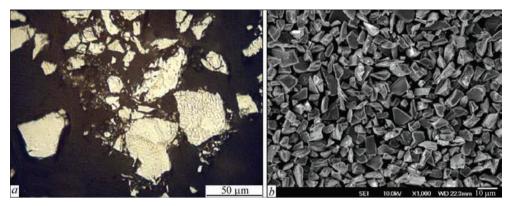


Figure 1. Microstructure of FeTi powder (a) and appearance of SiC powder (b)

of 1500 rpm and a ball mass-to-powder mass ratio of 10:1 in an air environment for 5 h. To eliminate sticking of the processed charge to the grinding bodies and walls of the drum, as well as to intensify the process of synthesis of new phases, a surfactant, oleic acid was added to the mixture ($C_{17}H_{33}COOH$) in the amount of 0.5 wt.%.

Coatings of CP FeTi-SiC were deposited by thermal spraying methods using both subsonic and supersonic high-temperature jets, which differ in the level of kinetic and thermophysical characteristics: subsonic method — plasma spraying (PS) and supersonic method — detonation spraying (DS). Plasma spraying was carried out in the "UPU-8M" installation using a mixture of Ar-N, as a plasma-forming gas. The detonation coating was applied in the "Perun-S" installation using a mixture of air-O₂-propane + butane as a combustible gas and with a cycle frequency of 6.6 s⁻¹. The spraying modes are given in Table 1. The technological spraying parameters were selected based on previous studies on spraying coatings using these methods from composite powders produced by the MChS method.

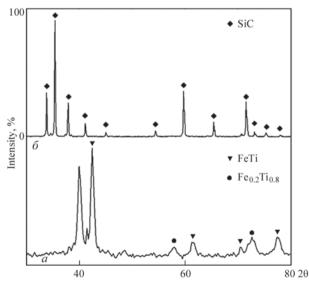


Figure 2. X-ray diffraction patterns of initial powders: a — FeTi; b — SiC

The study of CP and coatings produced from these powders was carried out using a comprehensive methodology that included metallographic, X-ray diffraction (XRD) and microdurotometric analysis (PMT-3 microhardness tester, at an indenter load of 25 g when investigating powder and 50 g for coating study).

X-ray phase analysis (XRD) was performed using a "DRON-3" diffractometer in $\text{Cu}K_{\alpha}$ -radiation with a graphite monochromator at a step movement of 0.1° and an exposure time of 4 s at each point with the subsequent computer processing of the obtained digital data. The phase identification was performed using the international ICDD PDF-2 or PDF-4 database. The size of crystallites in the coatings was estimated by the Debye–Scherrer formula:

$$d = \frac{K\lambda}{\beta\cos\theta},$$

where d is the average size of coherent scattering regions (domains, crystallites), which can be lower or equal to the grain size; K is the dimensionless particle shape factor (Scherrer's constant); λ is the X-ray radiation wavelength; β is the reflex width at half-height (in radians and in units of 2θ); θ is the diffraction angle (Bragg's angle).

The porosity of the coatings was determined using the Image-Pro Plus software.

The micromechanical characteristics of the surface of thermal coatings were determined by the microindentation method using a "MICRON-GAMMA" nanoindentometer [19]. The values of the characteris-

Table 1. Spraying modes of FeTi-SiC system coatings

Spraying method	Spraying mode						
	Current,	Voltage, V	Flow rate of working gases, m ³ /h	Spraying distance, mm			
PS	500	50	$Ar - 0.7$; $N_2 - 0.56$	100			
DS	-	-	Propane-butane -0.5 ; O ₂ - 1.55; Air - 0.65	100			

tics were calculated automatically according to ISO 14577-1:2015.

The aim of the studying coatings by the microindentation method was to:

- determine the structural state of plasma and detonation coatings produced from MChS FeTi–SiC powders;
- determine (based on the data on the mechanical properties of coatings) values of criteria that evaluate the behaviour of coatings under wear processes.

RESULTS OF THE STUDY AND THEIR DISCUSSION

In order to determine the phases that can be obtained in the FeTi–SiC system, the change in the value of the isobaric-isothermal potential (Gibbs energy) ΔG_T^0 was evaluated for the reactions of silicon carbide with simple elements (Ti, Fe). In the TS process, powder particles entering a high-temperature jet are significantly heated and, therefore, thermodynamic calculations were performed in the temperature range of 298–3000 K [14–16]. The following types of interaction were considered in the Ti and Fe–SiC systems (Figures 3, 4):

- 1. formation of a mixture of carbide and silicide phases of titanium or iron;
- 2. formation of titanium carbide, iron carbide and precipitation of free silicon;
- 3. formation of titanium silicides, iron silicides and precipitation of free carbon.

As is seen, for the Ti–SiC system, from a thermodynamic point of view, reaction *1* in the temperature range of 298–2100 K and reaction *2* in the tempera-

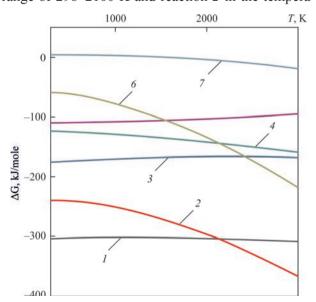


Figure 3. Temperature dependence of the change in the Gibbs energy of the reaction of silicon carbide with Ti: I — SiC + 8/3Ti = = 1/3Ti $_5$ Si $_3$ + TiC; 2 — SiC + 2Ti = TiC + TiSi; 3 — SiC + 3/2Ti = = TiC + 1/2TiSi $_2$; 4 — SiC + 5/3Ti = 1/3Ti $_5$ Si $_3$ + C; 5 — SiC + Ti = = TiC + Si; 6 — SiC + Ti = TiSi + C; 7 — SiC + 1/2Ti = TiSi $_2$ + C

ture range of 2100–3000 K are the most favourable (Figure 3). In the Fe–SiC system, reaction I in the temperature range of 298–680 K, reaction 2 in the temperature range of 1060–1180 K, and reaction 3 in the temperature ranges of 680–1020 and 1640–3000 K are the most favourable (Figure 4).

Thus, the following reactions are possible in the FeTi–SiC system:

SiC+
$$\frac{12}{7}$$
FeTi = TiC+ $\frac{1}{7}$ Ti₅Si₃+ $\frac{4}{7}$ Fe₃Si; (1)

$$SiC + \frac{3}{2}FeTi = \frac{1}{4}TiC + \frac{1}{4}Ti_{5}Si_{3} + \frac{1}{4}Fe_{3}Si + \frac{1}{4}Fe_{3}C + \frac{1}{2}C;$$
(2)

$$SiC + \frac{4}{3}FeTi = TiC + \frac{1}{15}Ti_5Si_3 + \frac{4}{15}Fe_3Si;$$
 (3)

$$SiC + \frac{3}{2}FeTi = \frac{3}{4}TiC + \frac{3}{4}TiSi + + \frac{1}{4}Fe_3Si + \frac{1}{4}Fe_3C.$$
 (4)

Calculations of the change in the value of the isobaric-isothermal potential showed that reactions *I* and *4* are the most favourable in the FeTi–SiC system from a thermodynamic point of view (Figure 5). At the same time, the content of silicon carbide in the mixture should amount to 21 wt.%. Therefore, a powder mixture of the composition 79FeTi–21SiC (wt.%) was used for the research. The final phase composition of both the MChS product as well as coatings may differ from the calculated one due to the non-equilibrium of

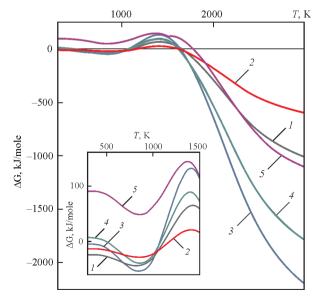


Figure 4. Temperature dependence of the change in the Gibbs energy of the reaction of silicon carbide with Fe: $I - \text{SiC} + 3\text{Fe} = \text{Fe}_3\text{Si} + \text{C}$; $2 - \text{SiC} + 5/3\text{Fe} = 1/3\text{Fe}_5\text{Si}_3 + \text{C}$; $3 - \text{SiC} + 6\text{Fe} = \text{Fe}_3\text{Si} + \text{Fe}_3\text{C}$; $4 - \text{SiC} + 14/3\text{Fe} = 1/3\text{Fe}_5\text{Si}_3 + \text{Fe}_3\text{C}$; $5 - \text{SiC} + 3\text{Fe} = \text{Fe}_3\text{C} + \text{Si}$

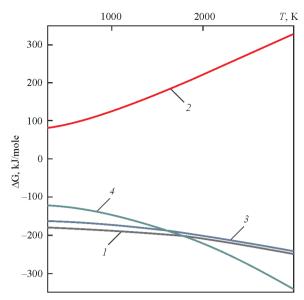


Figure 5. Temperature dependence of the change in the Gibbs energy in the reaction of ferrotitanium with silicon carbide (curve numbers correspond to the numbers of FeTi + SiC reactions)

the MChS and thermal spraying processes (high heating and cooling rates), which affects the completeness of the reactions of the initial components.

During the MChS of the powder mixture 79FeTi+21SiC (wt.%), the initial components are grinded with their simultaneous mixing, introduction of silicon carbide particles into the volume of ferrotitanium with the further "cold" welding of particles and formation of agglomerates. As a result of MChS, after 5 h of treatment, a composite powder with a heterogeneous fine-dispersed structure with an average particle size of $\sim 12~\mu m$ is formed (Figure 6).

The X-ray diffraction pattern of the produced CP (Figure 7) shows that during the MChS process, the X-ray lines broaden with the appearance of "halos" in the range of angles $2\theta = 34-52^{\circ}$, indicating a grinding of the crystallite size (up to 30 nm) and partial amorphization of the powder structure. Analysing the phase composition of CP, it should be noted that the X-ray diffraction lines corresponding to the Fe_{0.2}Ti_{0.8} phase are absent in the X-ray diffraction pattern and the lines corresponding to the TiC and Ti₅Si₃ phases appear. The absence of diffraction lines of the Fe_.Si compound, the

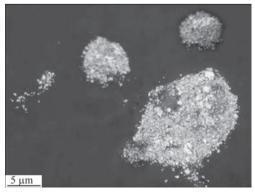


Figure 6. Microstructure of CP FeTi-SiC after 5 h of MChS

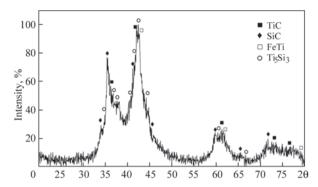


Figure 7. X-ray diffraction pattern of FeTi–SiC powder after 5 h of MChS

formation of which is possible according to equation 1, may be the result of the appearance of an amorphous halo, which complicates its identification. On the other hand, for the mechanically activated self-propagating synthesis in the MChS process, the value of the enthalpy-to-heat capacity ratio ($\Delta H/C_p$) should be greater than 2000 K [17]. Calculations have shown that for TiC, this ratio is 5400 K, for Ti₅Si₃ — 3100 K, and for Fe₃Si — 950 K. Therefore, the formation of iron silicide is thermodynamically less favourable, than the formation of TiC and Ti₅Si₃ compounds.

As a result of the formation of an amorphous nanocrystalline structure consisting of a FeTi matrix with dispersed inclusions of solid SiC, TiC, and Ti_5Si_3 phases, the microhardness of the particles of the produced CP is 8.2 ± 1.6 GPa.

One of the main requirements for the uniform supply of powder to a high-temperature jet during TS is its flowability. As a result of particle size grinding, in the MChS process, the specific surface area of the powder increases, which in turn reduces its flowability [18]. In order to provide flowability and optimize the particle size distribution of CP, before spraying, it was conglomerated with a 5 % aqueous solution of polyvinyl alcohol, followed by grinding and screening of particles, whose size falls in the range of 40–80 µm.

The X-ray diffraction phase analysis (Figure 8) revealed that the phase composition of the sprayed coatings is slightly different from that of CP FeTi-SiC, which indicates structural transformations in the powder particles that occur during the passage of particles through a high-temperature jet and during the formation of the coating layer. The degree of amorphousness of the coatings decreases relative to CP and the size of crystallites increases to 140 and 70 nm for plasma and detonation coatings, respectively. The absence of the SiC phase in the X-ray diffraction patterns also indicates the occurrence of phase transformation processes during spraying. In addition to the TiC and Ti_sSi_s phases present in the phase composition of CP, phase transformations in the coatings result in the formation of the Fe, Ti phase instead of FeTi, and in the case of plasma spraying, the Fe, TiSi Heusler phase

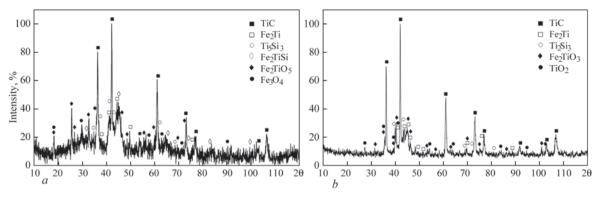


Figure 8. X-ray diffraction patterns of CP FeTi–SiC coatings produced by plasma (a) and detonation (b) spraying

is formed in the coating. During the presence of CP particles in a high-temperature jet, during spraying, they interact with air oxygen and oxidise the CP components, resulting in the formation of oxide phases in the coatings: pseudobrookite Fe_2TiO_5 and iron oxide Fe_3O_4 in the case of plasma spraying (Figure 8, a), ulvospinel Fe_2TiO_4 and titanium oxide TiO_2 in the case of detonation spraying (Figure 8, b).

As a result of plasma spraying, coatings with a heterogeneous structure and distinct structural components are formed (Figure 9, a). Defects in the structure of the plasma coating in the form of discontinuities and chipping of various shapes and sizes were found, the amount of which in the coating is \sim 13 vol. %. The size of defect ranges from 10 to 50 µm and their location across the coating cross-section is uneven. The presence of defects in the coating in the form of discontinuities is a consequence of insufficient thermal and kinetic energy of particles during spraying, which leads to a decrease in the impact consolidation and the degree of deformation of particles during the layer formation, on which the density of the coating depends. The size of defects of up to 50 µm indicates the presence of conglomerate-type particles in the powders after passing through the plasma jet, which are formed as a result of coagulation and fusion of particles of the conglomerated powder. Such particles do not undergo a melting stage and do not deform during the formation of the coating layer, and therefore the particles are not fixed in the coating due to the lack of a strong bond between the sprayed particles. As a

result, coatings with low cohesive strength are formed and defects in the form of chipping are formed in the coatings when producing microsections. It is possible to reduce the probability of such defects in the coating by optimising the process of plasma spraying of the FeTi–SiC composite powder by changing the technological parameters of plasma spraying.

The detonation spraying of CP FeTi-SiC results in the formation of a coating with a thin-lamellar heterogeneous structure. Small amounts of partially deformed particles are present in the coating. The oxides in the coating have the form of thin lamellae, located uniformly across the entire cross-section of the coating. The porosity of the coating is \sim 5 %. The absence of defects in the detonation coating, which are present in plasma coatings, is explained by the higher gas flow rate during the spraying process and, accordingly, the higher velocity of sprayed particles, which can reach ~800 m/s, while in plasma spraying, the maximum particle velocity can reach 200 m/s. Therefore, particles in detonation spraying acquire higher kinetic energy, which increases the degree of their deformation during the layer formation, resulting in the formation of dense coatings.

The microhardness of the coatings amounts to 8.5 ± 2.5 GPa in plasma spraying and 8 ± 2 GPa in detonation. The large variation in the measured microhardness values (6–11 GPa for plasma coating and 6–10 GPa for detonation coating) is explained by the heterogeneity of the structure of the coatings, composed of a metal component, carbide, silicide and

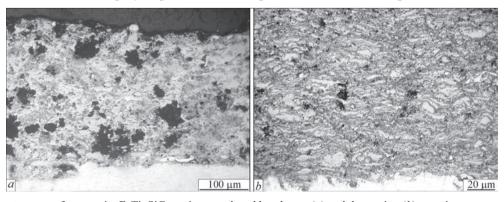


Figure 9. Microstructure of composite FeTi–SiC coatings produced by plasma (a) and detonation (b) spraying

Table 2. Mechanical properties of FeTi-SiC thermal coatings

Spraying method	Mechanical characteristics							
	$H_{\rm IT}$, GPa	E, GPa	E*, GPa	ε _{es} , %	σ _{es} , GPa	$H_{\rm IT}/E^*$	H^{3}_{IT}/E^{*2}	H^2_{IT}/E^*
PS	6±02	102±2.3	98	1.87	1.84	0.061	0.022	0.37
DS	5.2±0.5	93±10	89.2	1.78	1.65	0.058	0.018	0.32

oxide phases. An increase in the microhardness of the plasma coating relative to CP is associated with the formation of the Fe₂TiSi phase in the coating, the hardness of which can reach from 19 to 30 GPa [20, 21]. A slight decrease in the microhardness of the detonation coating relative to CP is associated with the formation of a significant amount of oxide phases in the coating, which occurs due to the oxidation of powder components during the spraying process.

As a result of the studies carried out by the microindentation method, a number of characteristics of the mechanical properties of the outer layer of plasma and detonation FeTi–SiC coatings were obtained:

 $H_{\text{\tiny IT}}$ — material hardness at indentation, GPa;

E — modulus of elasticity, GPa;

 E^* — effective (contact) modulus of elasticity, GPa;

 ε_{es} — relative out-of-contact elastic strain, %;

 σ_{es} — stress of out-of-contact elastic deformation, GPa;

 H_{II}/E^* — index of transition from elastic deformation to fracture or normalized hardness;

 H_{II}^3/E^{*2} — index of resistance to plastic deformation:

 $H_{\rm IT}^2/E^*$ — parameter of elastic recovery, i.e. the ability of the material to absorb strain energy.

The results of microindentation of coatings are shown in Table 2.

The analysis of the obtained data shows that according to the main indices of mechanical characteristics, the coating produced by detonation spraying is somewhat inferior to the coating produced by plasma spraying, which is associated with a higher content of oxide components in detonation coatings. It should be noted that the difference in the values of microhardness and hardness obtained by microindentation (nanohardness) is explained by the difference in measurement procedures. The load during microindentation is much lower and therefore the imprints are limited to the area of a single deformed particle and reflect its properties. Whereas the results of microhardness measurement reflect the average value of the coating.

According to the method for evaluating the structural state of a material using the dependence of the hardness value ($H_{\rm IT}$) on the value of the relative out-of-contact elastic strain ($\varepsilon_{\rm es}$), proposed by the authors of [22], FeTi–SiC coatings produced by both plasma and detonation spraying, belong to the group of microcrystalline materials with a heterophase structure.

According to the indices of $H_{\rm IT}/E^*$, $H^2_{\rm IT}/E^*$, $H^3_{\rm IT}/E^{*2}$, used as criteria for the wear resistance of strengthening coatings [23], the detonation coating is 1.1–1.2 times inferior to the plasma coating. Based on this, it should be expected that under sliding friction conditions, the FeTi–SiC coating produced by plasma spraying will have higher wear resistance than the coating of the same composition produced by detonation spraying.

CONCLUSIONS

On the basis of thermodynamic calculations of reactions between ferrotitanium and silicon carbide, the most favourable reaction in terms of interaction of components was established and the composition of the mixture of powders of the initial components was determined to produce a composite powder by the method of mechanochemical synthesis, at which the content of silicon carbide amounts to 21 wt.%. It was found that in the process of mechanochemical synthesis, a composite powder with an amorphous-nanocrystalline structure is formed, the phase composition of which, due to the non-equilibrium of the MChS process, differs slightly from the calculated one and consists of a FeTi metal matrix with dispersed inclusions of solid SiC, TiC and Ti_sSi_s, phases in it. The microhardness of particles is 8.2 ± 1.6 GPa.

As a result of thermal spraying, coatings with a heterogeneous structure were produced consisting of a Fe₂Ti metal matrix with ceramic inclusions of titanium carbide (TiC) and titanium silicide (Ti₅Si₃) and oxide phases: Fe₂TiO₅ and Fe₃O₄ in the case of plasma spraying, Fe₂TiO₄ and TiO₂ in the case of detonation spraying.

It was found that when using the detonation method of spraying FeTi–SiC powder, denser coatings with a porosity of \sim 5 % are formed, whereas during plasma spraying, the number of defects in the form of discontinuities and chipping is \sim 13 %, which is associated with low cohesive strength of the coating. In turn, detonation coatings have somewhat lower values of microhardness and mechanical characteristics (by 1.0–1.2 times).

Using the method of microindentation, it was found that, according to their structural state, the produced coatings of the FeTi–SiC system belong to the group of microcrystalline materials with a heterophase structure in terms of hardness and relative out-of-contact elastic strain, which are $H_{\rm IT}=6$ GPa, $\varepsilon_{\rm es}=1.87$ % for the coatings produced by plasma spraying, and $H_{\rm IT}=5.2$ GPa, $\varepsilon_{\rm es}=1.78$ % for the coatings produced by detonation spraying.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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