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Microstructure-related properties of explosively welded multi-layer Ti/ Al composites after rolling and annealing

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Abstract

The processes of rolling and annealing of explosively welded multi-layered plates significantly affect the functional properties of the composite. In current research, fifteen-layered composite plates were fabricated using a single-shot explosive welding technique. The composites were then rolled up to 72% to reduce layer thickness, followed by annealing at 625 °C for varying times up to 100 h. Microstructure evolution and chemical composition changes were investigated using scanning electron microscopy equipped with energy-dispersive spectroscopy. The mechanical properties of the composite were evaluated by tensile testing, while the strengths of individual layers near the interface were evaluated by micro-hardness measurements. After explosive welding, the wavy interfaces were always formed between the top layers of the composite and the wave parameters decreasing as the bottom layers approach. Due to the rolling process, the thickness of Ti and Al layers decreases, and the waviness of top interfaces disappeared. Simultaneously, the necking and fracture of some Ti layers were observed. During annealing, the thickness of layers with chemical composition corresponding to the Al₃Ti phase increased with annealing time. A study of growth kinetic shows that growth is controlled by chemical reaction and diffusion. The results of micro-hardness tests showed that after annealing, a fourfold increase of hardness can be observed in the reaction layers in relation to the Ti, while in relation to Al, the increase of hardness is even 15 times greater.

Keywords Multi-layer Al/Ti composite \cdot Explosive welding \cdot Hot-rolling \cdot Annealing \cdot Al₃Ti phase

1 Introduction

Parts in combat vehicles manufactured from homogeneous or multi-layer high-strength steel plates [1] are characterized by high specific gravity [2], which, in some cases can limit their applicability [3]. Therefore, research is being conducted at several research centers, looking into the possibility of using metallic materials that offer both high ballistic protection and the lowest possible specific gravity [4]. Since advanced armor usually consists of multiple layers of different materials that play a specific role in impact energy absorption, there are two ways to achieve the desired armor properties [5]. One way is through the combined use of ceramics, plastics, and/or fiber-reinforced epoxy resins [6]. Another way is the construction of the layered composites based on high-strength light metals, like aluminum/steel [7], magnesium/aluminum/titanium [8] or aluminum/aluminum/titanium [9]. The latter solution proposed by Godzimirski et al. [10], Tasdemirci et al. [11] and Płonka et al. [12] includes composite-like multi-layer plates, mostly based on titanium (Ti) and aluminum (Al). Ti and Al and their alloys, as structural materials, attract attention due to their low density, high stiffness and strength-to-weight ratios, corrosion resistance, and high-temperature resistance (Ti) or good thermal conductivity (Al). Therefore, titanium–aluminum-based composites open up many new possibilities.

Welding of the Ti- and Al-based alloys is difficult due to the strong difference in melting points and very high reactivity concerning nitrogen and oxygen at high temperatures. Currently, there are many methods that can be used for the multi-layered Ti/Al composite fabrication through solid-state joining processes, e.g., using

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Chemical com	position (wt.	. %)										
Alloy	J	z	0	Н	Cu	Mg	Si	Fe	Mn	Zn	Τi	Al
li Gr.1	0.08	0.03	0.18	0.015	I	I	I	0.2	I	I	Bal.	I
41 AA1050	I	I	I	I	0.05	0.05	0.25	0.4	0.05	0.07	0.05	Bal.
Mechanical pro	operties											
	Hardnes	s (HBW)		Tensile Strength (MP.	a)	Y	ield Strength (MPa			Modulus of Elasticity	y (GPa)	
li Gr. 1	120			240		1	70			105		
A1 AA1050	33			145		8	4			68		





Fig. 1 Scheme for Ti(Gr. 1)/Al(AA1050) multi-layer EXW

accumulative roll-bonding (ARB) [13], hot-rolling [8], hot-pressing [14], or explosive welding (EXW) [15]. During ARB process, to reduce deformation resistance, it is necessary to isothermally heat-treat plates prior to the rolling. Thus, hot roll-bonding technology is a high-cost and overly complex process. Hot pressing seems to be a much more promising method in contrast to ARB process. However, this process enables to produce composites with limited size, determined by the size of the press machine. To produce a large-scale product, EXW method is successfully used. This method enables to join of a wide range of materials with different properties that are difficult or impossible to join by other methods. So far, several studies have described the various combinations of composites produced by EXW technique, such as Al/Cu proposed by Lokal et al. [16] and Paul et al. [17], Al/steel [18], Al/Mg proposed by Mroz et al. [19], Zeng et al. [20] and Paul [21], steel/Cu proposed by Dyja et al. [22], Andreevskikh [23] and Paul et al. [24], Al/Ti proposed by Bataev [25] and Paul [26], and many others.

Because no impurities are observed at the interfaces in multi-layer plates produced via explosive welding, migration of easy atoms to the adjacent layers occurs during postwelding heat treatment. (As shown by Bataev et al. [25], the dynamic of the diffusion in explosively welded composites is more than 3 times higher than in clads produced using the hot-pressing method). The structure modification via initiation of the diffusion processes leads to the formation of hard intermetallic layers. TiAl, Ti₃Al, and Al₃Ti phases are the most favorable intermetallic compounds in the Ti–Al binary systems. Zhang et al. [27] have shown that Al₃Ti phase has the lowest effective free energy, so it is easier to form at this interface. The Al₃Ti phase is of interest due to its light weight, oxidation resistance at high temperatures, relatively high Young modulus (216 GPa), and low density (3.3 g/ cm³), which is lower than in Ti and Al-based compounds [28]. A strong increase in the performance of the composite can therefore be expected by introducing hard intermetallic layers based on the Al₃Ti phase into the structure.

The aim of the current research is to investigate the phenomena that occur at the interfaces of fifteen-layered Al/ Ti composite. Special attention was paid to the dynamic of the intermetallic phase growth leading to the disappearance of the soft (Al) components. Single-shot EXW process was used to produce multi-layered composite, whereas hot-rolling was applied to reduce the thickness of individual layers. Finally, the heat treatment at 625 °C for times ranging between 1 and 100 h was applied to induce intermetallic phase growth. Subsequently, analysis of the growth dynamic was performed to determine the factors controlling the quality of interfacial layers. The analyses were conducted using scanning electron microscopy (SEM) equipped with backscattered electron (BSE) and X-ray energy-dispersive spectrometry (EDS) detectors. The microstructural analyses were correlated with micro-hardness measurements.

2 Materials and methods

2.1 Materials and explosive welding

The tested plate was composed of eight sheets of Ti Gr. 1 (hereafter denoted as Ti) and seven sheets of AA1050 alloy (hereafter denoted as Al). The sheets' size was 210 (length) \times 300 (width) mm². The materials of the sheets in the initial state were characterized by a uniform, fully recrystallized microstructure. The chemical composition and mechanical properties of Ti and Al alloys are given in Table 1. The thickness of the Ti and Al sheets was 1 mm.

EXW was conducted by High Energy Techniques Works 'Explomet' (Opole, Poland). The single-shot explosion welding technique was applied to form a fifteen-layered plate composed of alternatively arranged Ti and Al sheets. In the applied set-up, the top and bottom layers were always Ti. The composites were obtained using the constant stand-off explosive cladding technique with an initial 1 mm standoff distance between the layers, 5 mm thick flyer sheet, and steel anvil. Before welding, the contact surfaces of the joined sheets were ground, cleaned of solid contaminants, and degreased. A detonator was placed in the middle of the



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Fig. 3 View of the samples after the first pass $({\bf a})$ and after the second pass $({\bf b})$

 Table 2
 Sample height and applied reduction of the composite during hot-rolling

Pass no	sample height [mm]	height reduction per pass [%]	total height reduction [%]
0	12.8		
1	7.0	45	45
2	3.6	49	72

shorter edge of the flyer plate. A modified Saletrol explosive charge with a layer thickness of 30 mm was used to initiate a detonation velocity of 2320 m/s. An Explomet-Fo-2000 measuring instrument (Kontinitro) was used to measure the detonation velocity. It is an electronic timer for which light signals initiate and stop measuring. The scheme for Ti/Al multi-layer single-shot EXW is presented in Fig. 1.

2.2 Hot-rolling and annealing processes

The EXW plates were then hot-rolled using a semi-industrial rolling mill (D300 mm). The thermoplastic processing was performed in two passes at 430 °C up to a total thickness reduction of 72%. The rolling speed was 0.2 m/s. For the rolling process, the specimen with dimensions of $13 \times 30 \times 100$ mm³ was cut from the EXW plate. The samples were prepared in a way that the rolling direction (RD) was parallel to the detonation direction (DD). Materials before rolling were annealed in a laboratory furnace at 450 °C for 20 min, then the annealed material was quickly transferred to the rolling mill. The time between transferring material from the furnace to the rolling mill was less than 5 s. The multi-layer feedstock was heated in the LAC KC 120/14 resistance chamber furnace before the individual passes. After the rolling process, the samples with dimensions of $15 \times 5 \times 2$ mm³ were cut off from the middle section of the rolled sheets. Then the samples were annealed at 625 °C for times ranging between 1 and 100 h using a furnace without a protective atmosphere.

2.3 Microstructure observation, tensile test and micro-hardness measurements

The samples for microscopic observations were cut along the DD from EXW plates and along RD from



Fig. 4 SEM/BSE image showing Ti/Al composite after hot-rolling



Fig. 5 SEM/BSE images showing the growth of reaction layer after hot-rolling and annealing at 625 °C for 1 h (a, b), 10 h (c, d), and 100 h (e, f)

post-annealed hot-rolled sheets, using a wire saw. The metallographic examinations of samples were carried out using an optical microscope (OM) and SEM (XL30 Philips) equipped with a BSE and EDS detectors. The tensile tests were carried out at room temperature (RT) using an Instron Model 6065 Universal Testing Machine with a strain rate of 0.00025 s^{-1} . The tensile samples were cut perpendicular to the rolling direction of the composite sheets. Dog-bone-shaped tensile specimen with a thickness 5 mm, a width of the test section of 3 mm, and the length of 40 mm was machined with the use of electrical discharge saw. Three tensile specimens were tested for each

type of materials (3 specimens after hot-rolling as well as 3 specimens after hot-rolling and annealing at 625 °C for 100 h). The ultimate tensile strength, yield strength, and elongation were determined from the stress–strain curves.

Vickers micro-hardness measurements were conducted on finely polished surfaces on ND/(DD or RD) sections to identify the micro-hardness of the reaction regions near the joint surface. The measurements were performed with a load of 100 G ($HV_{0.1}$) and a dwell time of 15 s. The obtained micro-hardness values were given as the average of three indentation measurements.



Fig. 6 Changes in the average thickness of the reaction layer at the top, middle, and bottom interfaces after annealing

3 Results and discussion

3.1 Macroscopic observations

Figure 2 shows the structure of the composite, in the aswelded state, typically observed in the ND/DD section. Strong variation of the interface morphology was observed across the clad thickness. The formation of the wavy interface between the first two (top) layers of the composite located near the flyer plate, i.e., close to the explosive charge, is well visualized (Fig. 2a). In contrast, the interfaces between the layers in the middle and at the bottom of the composite were almost flat (Figs. 2b, c). The changes in the morphology of the contacting layers were due to the formation of wave-type features reflecting turbulent flow of layers near the interface. The beginning of the first interface had the strongest waviness, whereas the interfaces between plates at the bottom part of the composite were nearly flat. The result of this observation corresponds to earlier studies by Bataev et al. [25], Paul et al. [26], and Petrzak et al. [29], who showed that clads produced by EXW perform significantly different in the interface morphology across the composite thickness and along its length. They also observed that the wave amplitude linearly depends on the kinetic energy loss due to the collision of the sheets. This energy rapidly decreases with each successive collision of the sheets. Morphological changes are also due to the occurrence of melted regions. In the upper layers, these reaction regions are mainly located in the wave vortexes and on the wave crests. In the middle and bottom parts of the composite, they take a form of (semi)continuous layers situated along the interfaces. However, a decreasing quantity of the reaction regions can be observed as the bottom surface of the composite is approached.

Figure 3 shows the structure of the EXW plate after a two-pass hot-rolling. After this process, the total thickness of the plate was reduced by 72% (Table 2) up to 3.6 mm. The thicknesses of individual layers were reduced up to 190–430 μ m and 210–420 μ m for Al and Ti, respectively, whereas the wave amplitude of the upper layers decreased significantly. Simultaneously, the discontinuities of the Ti layers were observed (Fig. 4). In the study of Assari and Eghbali [30], such discontinuities are associated with necking and subsequent crack formation of Ti layers; these phenomena are thought to be caused by the lower ductility of Ti as compared to Al. In the areas, where discontinuities of Ti layers occur, Al layers merge. This locally increases the thickness of Al layers up to 600–650 μ m. Since the specimen with an initial width-to-height ratio of 2.4 was used in

Fig. 7 SEM/BSE images showing discontinuities formation after hot-rolling and annealing at 625 °C for: **a** 20 h and **b** 100 h





Fig. 8 The plot of *ln w* versus *ln t* by linear fit analysis

the rolling process, an intense plastic flow of the individual layers in both the RD and TD sections was observed. This was especially well observed for the Al layers, which were extruded intensively from between the Ti layers (Fig. 3).

4 The dynamic of the Al₃Ti phase growth

The kinetic of the intermetallic phase growth during heat treatment of EXW and/or hot-rolled composites has been the subject of many studies, e.g., Lazurenko et al. [15], Petrzak

et al. [29] and Fronczek et al. [31]. Most of them were conducted at temperature ranges between 550 and 640 °C. The results of these tests vary, depending on the thickness of the sheets and bonding parameters, such as detonation velocity or stand-off distance, between layers applied during the process.

Figure 5 shows the SEM/BSE microstructure of hot-rolled Ti/Al sheets after heat treatment at 625 °C and for annealing times ranging between 1 and 100 h, while changes of the intermetallic layer thickness as a function of time are shown in Fig. 6. After the annealing time of 1 h, the thickness of the intermetallic layers reached ~ $1.9 \,\mu\text{m}$ and ~ $1.2 \,\mu\text{m}$ for the top (Fig. 5a) and bottom (Fig. 5b) interfaces respectively, whereas in the center of the composite, it is significantly smaller (~ $0.9 \mu m$). Extending the annealing time up to 2 h caused double increase in the thickness of the intermetallic layers, in all three areas tested. Uniformity of the thickness of the intermetallic layer across the composite thickness was observed in the range of annealing times up to 10 h (Fig. 5c, d), with an increase of the intermetallic phase up to $12 \,\mu m$ observed. Increase of annealing time to 100 h (Fig. 5e, f), contributes to significant increase of the width of the intermetallic layers. However, areas where the Al layers were not completely consumed were observed. This can be seen in Fig. 7b, which shows unreacted areas at the top and bottom layers of the composite, while the middle layers of Al are fully reacted. Comparing the microstructures in Figs. 4 and 7a, differences in the width of the Al layers in the different parts of the composite are observed; the Al layers in the



Fig. 9 Total width of the reaction layer formed upon annealing at 625 C for between 1 h and 100 h. The results are compared with those obtained by: Lazurenko et al. [15]; Bataev et al. [25]; Petrzak et al. [30]; Fronczek et al. [31, 34, 39]; Xu et al. [32] and Foadian et al. [36]

				duras un ase refer						
	Present paper	Petrzak et al. [2019]	Fronczek et al. [2018]	Fronczek et al. [2017]	Fronczek et al. [2016]	Fronczek et al. [2016]	Lazurenko et al. [2016]	Foadian et al. [2014]	Bataev et al. [2012]	Xu et al. [2006]
The temperature of annealing [°C]	625	600	630	630	550	630	640	630	630	630
Number of inter- faces	14	14	7	2	1	1	39	5	20	6
Detonation veloc- ity [m/s]	. 2320	2600	1900 ± 50	1900 ± 50	1900 ± 50	1900 ± 50	4200	4500	4200	Not specified
Annealing condi- tions	Air	Air, hot-pressing	Vacuum	Vacuum	Vacuum	Vacuum	Air	Ambient	Air	Vacuum
Mechanism of Al ₃ Ti phase growth		NS	SCR, VD and CR	VD and CR	QV	CR	SD	DF and CR	SN	DF
DF diffusion, SD	solely diffusion,	VD volume diffusio	m, CR chemical react	tion, SCR solely ch	remical reaction, N.	S not specified				

Table 4 Mechanical properties of the platers after hot-rolling and after hot-rolling and annealing at 625 °C for 100 h

Configuration	Rm [MPa]	R0.2 [%]	A [%]
Hot-rolling	331.21	303.16	20.4
Hot-rolling + annealing	105.55	94.06	13.0

upper and lower regions are much wider than in the middle region of the sample, Fig. 7a.

The SEM/EDS chemical composition measurements revealed the presence of layers consisting of 71% of Al and 29% if Ti, suggesting a formation of the Al₃Ti intermetallic phase. It was the only intermetallic compound formed independently during the applied annealing time. This result is consistent with previous work by Xu et al. [32] and van Loo and Rieck [33], who reported that Al₃Ti is the only phase that can be observed at the Ti/Al interface (Gibbs free energy for the formation of the Al₃Ti phase was substantially lower than those of other stable phases). Similar results on multi-layered composites were reported by Lazurenko et al. [15], Petrzak et al. [29] and Fronczek et al. [34], who documented that the heat treatment of Al/Ti composite led to the rapid growth of layers composed of the Al₃Ti intermetallic phase along the entire interface. It has also been documented in these works that the areas of primary melted regions composed of several non-equilibrium phases, i.e., not observed on Al-Ti equilibrium phase diagram, are transformed during heat treatment (via atoms migration) into the equilibrium Al₃Ti phase. Since the solubility of Al in Ti is 3 times greater than the solubility of Ti in Al, therefore Al layers are consumed significantly faster than Ti ones. Annealing time strongly influences the rate of the Al₃Ti phase growth. Dybkov [35] and Foadian et al. [36] described the growth of the Al₃Ti phase as a process controlled by two different mechanisms-diffusion and/or chemical reaction. The growth of the reaction layer during annealing can be expressed via the well-known equation $w = kt^n$ [37], which relates the width of the reaction layer (w) to the annealing time (t). The parameters k and n are constants. It is widely accepted that the value of *n* determines the mechanism responsible for the growth of the reaction layer; if n is close to 1, the growth process is controlled by chemical reactions, while if it is close to 0.5, the growth process is controlled by diffusion. The plot of $\ln(w)$ versus $\ln(t)$ is shown in Fig. 8.

In the range of annealing times analyzed, strong changes in the n value are observed. For annealing times up to 10 h, the value of n is close to 1, which suggests that growth of the Al₃Ti layer is controlled by a chemical reaction. This is mainly due to the small distance between the Al and Ti layers, which facilitates faster mutual diffusion across the interfaces. For annealing times above 20 h, the n value is close to 0.5, suggesting that the growth of the Al₃Ti layer is mainly



Fig. 10 Representative stress–strain curves obtained for samples after hot-rolling and after hot-rolling and annealing (**a**) as well as SEM/SE images showing fracture morphology of the sample after hot-rolling (**b**, **c**) and after hot-rolling and annealing at 625 °C for 100 h (**d**, **e**)

controlled by diffusion. The chemical reaction is hindered because it occurs through the already formed Al_3Ti layer.

The growth kinetics of the Al₃Ti phase in explosively welded composites has been the subject of many studies. Foadian et al. [38], Bataev et al. [25], Fronczek et al. [31, 34, 39] and Xu et al. [32] analyzed this aspect during the annealing at 630 °C. Studies of growth kinetics at 640 °C were carried out by Lazurenko et al. [15], whereas at 600 and 550 °C by Petrzak et al. [29] and by Fronczek et al. [31], respectively. Figure 9 and Table 3 present a change in values of the average thickness of the reaction layer measured in the current research and a comparison with previously obtained literature data. It can be concluded that different mechanisms are responsible for the growth of the reaction layers. It may be related with different parameters applied during bonding which results in strongly different microstructural changes near the interface. However, in majority of the analyses, the kinetic of growth occurred as result of volume diffusion or chemical reaction (Table 3). Study on growth of the reaction layer carried out at 550 °C by Fronczek et al. [31] showed that bulk diffusion was dominant. However, Thiyaneshwaran et al. [40] showed that, in addition to the diffusion mechanism, chemical reaction also plays a significant role.

To evaluate the mechanical properties of the clad and in particular intermetallic phase, the tensile stress and microhardness measurements were carried out.

Results of the tensile test for the hot-rolled and postannealed hot-rolled composite (annealing at 625 °C for 100 h) are summarized in Table 4, and representative stress–strain curves are shown in Fig. 10a. The general conclusion is that the hot-rolled composites show higher mechanical properties compared to the hot-rolled and annealed composites—maximum tensile strength (Rm) for the hot-rolled composite was 331.2 MPa, whereas for the hot-rolled and annealed composite was 105.6 MPa. Similarly, the yield strength values (R0.2) were 303.2 and 94.1 MPa, for the hot-rolled and post-annealed hot-rolled composite, respectively. The relative elongation (A5) for hot-rolled composites was 20.4%, while for annealed hot-rolled not-rolled ones, it was 13.0%.

Tensile tests for hot-rolled state revealed ductile character of fracture—(Fig. 10b, c), crack and numerous cavities are visible (Fig. 10c), forming a typical cup-and-cone surface. In contrast, brittle fractures were observed in the composites after hot-rolling and annealing at 625 °C for 100 h (Fig. 10d, e). Numerous delaminations were also observed in the interface areas between the Ti and Al₃Ti phases.

Micro-hardness measurements were performed on the longitudinal section across the interface and the results are shown in Fig. 11. In the 'as-welded' state, large micro-hardness gradients close to the interfaces, resulting from a strong strengthening of the jointed sheets, can be observed. The micro-hardness results range between 37 and 43 $HV_{0.1}$ for Al and 186 to 213 $HV_{0,1}$ for Ti. In the case of Al, the increased micro-hardness is limited to a thin layer close to the joint surface, while Ti undergoes strong strengthening over a much longer distance. The results of micro-hardness measurements for sample after 1 h annealing (Fig. 10b) showed a slight decrease in the hardness of the joined materials. The hardness values vary from 29 to 33 $\mathrm{HV}_{0.1}$ and from 158 to 181 HV_{0.1} for Al and Ti, respectively. On the other hand, significant increase of micro-hardness after longer annealing times is related to the presence of the intermetallic layers. After annealing for 100 h (Fig. 10d), micro-hardness



Fig. 11 Variation in the micro-hardness on the ND/DD sections after a EXW, rolling and annealing at 625 °C for b 1 h, c 20 h and d 100 h

measured in Al and Ti are ~ 180 HV_{0.1} and ~ 34 HV_{0.1}, respectively. Reaction region is characterized by microhardness values ranged between 550 HV_{0.1} and 677 HV_{0.1}. According to Hu et al. [41], the Al₃Ti phase possesses high hardness, which is related to high shear modulus and low bulk modulus. Nevertheless, these parameters may have an impact on voids formation, especially between two intermetallic faces to Ti/Al₃Ti-Al₃Ti/Ti (Fig. 5f and Fig. 7b).

5 Conclusion

The kinetics of the intermetallic phase growth in Al/Ti-based composites fabricated using explosive welding technology subjected to hot-rolling and annealing processes were analyzed in this study. A 72% thickness reduction of the fifteen-layered Ti/Al composite was achieved by hot-rolling in two passes. During subsequent heat treatment at 625 °C, the Al layers reacted with Ti, forming a reaction layer with a chemical composition corresponding to the Al₃Ti phase. SEM/BSE analyses of hot-rolled and then annealed samples for times ranging between 1 and 100 h showed the systematic growth of a continuous layer of the intermetallic phase along all the interfaces, leading to the formation of a Ti/Al₃Ti/.../Al₃Ti/Ti-type composite. It was documented that due to the annealing process carried out for 100 h, almost all Al layers reacted. However, insignificant amount of unreacted Al was still observed. The growth of the reaction layer is governed by a chemical reaction in the first stage and controlled diffusion in the second stage. This resulted

in linear growth kinetics in the first stage and parabolic in the second stage.

The results of micro-hardness tests showed that longterm heat treatment results in a fourfold increase of hardness within the reaction layers with respect to the Ti layer, while as compared to the Al, the hardness value is even 15 times greater. However, the tensile tests revealed a formation of delamination between the Ti/Al₃Ti interfaces which decrease mechanical properties of Ti/Al composite after annealing at 625 °C for 100 h.

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Data availability Data can be supplied upon request.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

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