Characterization of microstructure and selected properties of SnSbCu alloy after FSP

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Abstract

The paper presents the results of research on the microstructure and selected mechanical properties of the SnSbCu-bearing alloy after friction stir processing (FSP). The Whorl tool was used for modification; the process was carried out using two rotational speeds of the tool: 280 and 450 RPM and a constant linear speed of 355 mm/min. Microstructure studies were performed employing the techniques of light microscopy and scanning electron microscopy along with analysis of the chemical composition of micro-areas. Additionally, the phase composition was investigated by means of the X-ray diffraction method and statistical analysis of the precipitates present in the investigated alloy. In addition, hardness, flexural strength, and uniaxial compression tests were performed before and after FSP modification. It was proved that using FSP to modify the SnSbCu alloy promotes refinement and homogenization of the microstructure, as well as improvement of the flexural strength, whereas no changes in the hardness level were found.

Keywords Friction stir processing · SnSbCu alloy · Microstructure · Mechanical properties

1 Introduction

Alloys used to produce sliding bearing bushes should have appropriate mechanical properties, abrasion resistance, and good corrosion resistance. Materials that exhibit such

characteristics are tin-based bearing alloys. The microstructure of these alloys is multi-phase, most often in the three-phase form: α , β , η or α , β , ε , where: α —an antimony and copper solution in tin, which is the soft and ductile matrix, β —angular SnSb crystals, and η —CuSn compound acicular precipitates [1, 2]. Due to the growing requirements for bearing alloys such as increasing loads/circumferential speeds and reducing machine sizes, the requirements for the strength properties are also rising. The data presented in scientific papers on tin Babbitts indicate improvement in the properties of these alloys as a result of refinement and even distribution of the hard support phases in the tin-rich soft matrix. The authors of work [3] proved that the change in the morphology and size of the intermetallic phases obtained thanks to Babbitt heat treatment promotes improvement of the mechanical properties. This means that the dispersion of secondary phase particles seems to be an effective mechanism to strengthen tin alloys for sliding bearings. Cold hardening of bearing alloys is almost impossible due to their low recrystallization temperature. The results of the work by Sadykov [4] show that applying plastic deformation in the rolling process at room temperature contributed to a significant increase in elongation in relation to the cast alloy (from 5 to 30%), with a simultaneous drop in the strength properties. This clearly indicates the occurrence of changes in the structure of the studied alloy related to

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recrystallization. Moreover, the refinement and change in the morphology of the hard SnSb intermetallic phases in particular improves the tribological properties. Barykin et al. [5] showed that the degree of refinement of β phase particles (SnSb) influences the wear intensity. The results of tests of the tribological properties under the conditions of technically dry friction presented by the authors proved that the wear intensity of the tested bearing alloy decreased by 25% after the heat treatment process and obtained by plasma spraying by 40%, compared to the alloy in the cast state. The positive effect of changing the morphology of the SnSb precipitates on the reduction in tribological wear was also presented in work [6]. Large, angular precipitates of SnSb with sharp edges easily fall out of the matrix, leaving voids behind and creating deep scratches during operation of the bearing, at the same time accelerating its wear. The authors of work [7] presented the positive influence of thermoplastic processing on the Babbitt properties. As a result of applying pressure (5 MPa) in the crystallization process and then using die forging at the temperature of 210–235 °C, they obtained an anti-friction layer with a fine-grained microstructure and evenly distributed β phase (SnSb) precipitates, which also contributed to reduction in the wear intensity. Potekhin et al. [8] tested various casting methods, including the turbulent casting method developed by the authors of the cited work, which made it possible to obtain a tin bearing alloy with the best microstructure with globular intermetallic phase precipitates and the most favorable tribological properties. The results concerning the improvement of the properties of tin matrix bearing alloys as a result of surface treatment can be found in [9, 10]. Dong et al. [9] proved that applying the process of laser remelting of the tin Babbitt surface promotes homogenization and refinement of the microstructure as well as improvement of the tribological properties, including a reduction in the wear rate of this alloy under lubricated friction conditions, at 25–100 °C. Due to the rapid cooling during laser remelting, the grain growth was inhibited, and the microstructure of the bearing alloy on the tin matrix was homogenized, which translated into an increase in the hardness of this alloy and a decrease in the coefficient of friction. Similarly, the authors of work [10] used laser surface texturing in order to improve the tribological properties of the Babbitt. The results of the tribological tests under lubricated friction conditions presented by the authors proved the reduction in the coefficient of friction of the alloy with the textured

surface. In addition, they indicated that surface texturing can play a key role in improving abrasion resistance.

Friction stir processing (FSP) is one of the innovative and promising methods of local modification of the microstructure of the surface layer [11, 12]. A significant advantage of this processing technique is the possibility of obtaining a fully recrystallized microstructure, characterized by equiaxed, fine grains in the weld nugget, which is formed by intensive plastic deformation at an elevated temperature, not exceeding the melting point [13]. Initially, the FSP process was used to modify the microstructure of light metal alloys such as aluminum and magnesium in order to increase their susceptibility to plastic deformation. Over time, increasingly more applications of the FSP process appeared, e.g., for modifying the microstructure of casting alloys or composite materials. The use of FSP to modify casting alloys results in homogenization and refinement of the microstructure, the removal of porosity, improvement of the mechanical properties, and increased fatigue strength, as well as abrasion resistance [14–16]. The results of the studies by Ma et al. presented in paper [14] showed that FSP modification carried out on the A356 aluminum alloy, and AZ91 magnesium alloy contributed to improvement of the mechanical properties as a result of the breakdown of coarse dendrites and secondary phases, refinement of the matrix grains, in addition to the dissolution of precipitates, and elimination of porosity. In addition, the authors noted that this treatment method is ideally suited for casting alloys containing coarse particles of secondary phases because it gives the possibility of supersaturation. Similar results of research on the microstructure after friction modification (FSP) of the surface of the A356 casting alloy were also obtained by the authors of works [15, 16] in the following years. The refinement and change in the morphology of the thick Si needles, as well as their even distribution in the matrix, contributed to an increase in hardness and a decrease in the coefficient of friction in addition to a decrease in abrasive wear of the tested alloy under conditions of technically dry friction [15]. Moreover, Alidokht et al. [16] proved that the increase in

Table 1 Chemical composition of investigated alloy, wt%

Name of alloy		Chemical composition, wt%			
Grade	Designation	Sn	Pb	Sb	Cu
SnSb9Cu4	B89	89.45	0.17	6.86	3.52

Fig. 1 Diagram of FSP modification of surface layer; figure also shows diagram of Whorl tool

the rotational speed of the tool during FSP modification resulted in an increase in the wear resistance in dry friction conditions. This phenomenon results from the refinement of the microstructure, and more precisely the load-bearing effect of the fine and almost spherical Si particles after FSP, which significantly reduces the shear stresses. The FSP technology also shows high efficiency in the homogenization of composites obtained by powder metallurgy, as proved by Berbon et al. [17]. The results of the research presented by the authors showed that FSP modification of aluminum composites (Al-Ti-Cu and Al-Ti-Ni) contributed to an increase in elongation (over 10%), while maintaining high tensile strength (approx. 650 MPa). An analysis of the FSP literature shows that frictional modification to a large extent has developed towards the production of surface

composites. The use of FSP treatment for the production of SiC-reinforced composite layers on the aluminum surface in 2003 by Mishra [18] contributed to the development of various composites based on copper [19], titanium [20], and steel [21] alloys in the following years. The successful applications of FSP described above have shown that FSP is a metalworking technique that provides local modification and the ability to control the microstructure in the subsurface layers of the processed metal components. FSP is successfully used in the automotive and aerospace industries, where materials are required to have greater resistance to wear, creep, and fatigue. Thanks to continued research efforts and a better understanding of FSP, increasingly more applications of this method in the production, processing, and synthesis of metal materials will be found.

Fig. 2 Microstructure of SnSb9Cu4 alloy after FSP modification at 280 RPM; LM

Fig. 3 Microstructure of SnSb9Cu4 alloy after FSP modification at 450 RPM; LM

As demonstrated earlier, it is possible to change the properties of bearing alloys through heat treatment, plastic deformation on the surface, and other modification methods. There are no data on the applicability of the FSP method to modify the surface layer of these alloys. The results concerning various materials such as aluminum alloys presented in the literature were so promising that an attempt was made to implement this method also for the most popular grades of tin-based bearing alloys. The results of the research on the use of the FSP method to modify the SnSb11Cu6 alloy were published in [22]. It has been proved, among others, that the FSP modification of the SnSb11Cu6-bearing alloy using the Triflute tool improves the tribological properties as a result of the refinement of hard phases present in the alloy. This article focuses on the characteristics of the

microstructure of the SnSb6Cu4 alloy containing, respectively, approx. 6 and 4 wt% antimony and copper after FSP modification using a Whorl tool.

The aim of the research was to determine the influence of the FSP process parameters on microstructural changes in the tool impact area, first of all, the possibility of increasing the dispersion of secondary phase particles, which in turn may translate into an improvement in the mechanical properties of this alloy.

2 Material and methodology

The material to be studied was the tin-based casting bearing alloy SnSbCu. The alloy was poured into cast iron molds, then

Fig. 4 Microstructure of SnSbCu alloy. **a, b** An initial state after casting. **c** Stir zone area 280 RPM, **d** Stir zone area 450 RPM, LM

cooled in air. The chemical composition of the studied alloy is presented in Table 1.

The melting temperature range of the alloy is 241–354 °C, density: 7.39 Mg/m³, yield point: Rp0.2 = 40 MPa (at the temperature of 20 °C) [23].

FSP modification was carried out on a welding stand built on an FYF32JU2 vertical milling machine. The alloy was subjected to FSP using a tool consisting of a spiral shoulder and a Whorl pin-cone-shaped with a spiral-shaped thread (Fig. 1). The tool is made of high-speed steel HS 6-5-2 (SW7M). FSP was conducted using two rotational speeds of the tool: 280 and 450 RPM and a constant linear speed of 355 mm/min.

Examination of the microstructure of the samples was performed by means of light microscopy (OLYMPUS GX51 microscope) and scanning electron microscopy along with EDS analysis of the chemical composition in micro-areas (Hitachi SU 70 microscope). Additionally, the phase

composition was analyzed using a BRUKER X-ray diffractometer with a Co K α = 0.179 nm (1.79 Å) cobalt lamp. Quantitative analysis of the precipitates was performed in ImageJ Fiji on the basis of SEM micrographs (in the BSE detector observation mode). The quantitative research included analysis of the size of the precipitates and the nearest neighbor distance (NND). Hardness measurements were made using the Brinell method with a hardness tester by Innovatest; a tungsten carbide ball with a diameter of 2.5 mm and a load of 31.25 kG were used. The average values of hardness and standard deviations were determined. The static uniaxial compression test was conducted at ambient temperature on a Zwick Roell Z020 testing machine. The compression test was carried out with a constant tool feed rate of 0.7 mm/s and was stopped after achieving a permanent deformation of 45%. Additionally, the Zwick Roell Z020 three-point flexural test was performed. The flexural test was

Fig. 5 Microstructure of B89 alloy after FSP modification with speed of 280 RPM and results of spot analysis of chemical composition, wt%; SEM

Pt.	Cu-K	Sn-L	Sb-L	Pb-M
1	0.22	55.42	43.52	0.84
2	0.20	98.20	1.48	0.12
3	38.94	59.48	1.58	-
4	0.08	55.39	0.86	43.68

Fig. 6 X-ray diffraction pattern of SnSbCu alloy for initial material and after FSP modification

conducted at room temperature and with a constant tool feed rate of 0.05 mm/s. In both the uniaxial compression and three-point flexural tests, the tests were performed on three samples from each variant.

3 Results and discussion

3.1 Microstructure characterization

Observations of the microstructure of the cross section of the zone after FSP modification depending on the rotational speed of the tool (Figs. 2, and 3) revealed a change in the structure and shape of the stir zone with an increase in the rotational speed of the tool. The FSP zone width and depth shrunk with increasing rotational speed. It is also worth drawing attention

to the limitation of the depth of the impact of the tool shoulder with the increase in the rotational speed, which is related to the change in the amount of heat introduced to the stir area and its temperature. Increasing the rotational speed of the tool results in the fact that the material just below the surface of the tool reaches a higher temperature, and thus is able to bear smaller loads; while becoming more plastic, it is “sheared” faster, and the depth of the shoulder impact is smaller. Previous studies show that the microstructure in the process zone depends on the process parameters (rotational speed of the tool, travel speed, pressure force), the type of material being processed, and the shape of the tool [24–26]. The state of knowledge regarding the phenomena occurring during FSP is not yet fully known, which makes it difficult to select the optimal parameters for various alloys. In addition, observations of the microstructure after FSP modification revealed the presence of a nugget in the center of the FSP zone, characterized by a lower degree of particle refinement, which is clearly noticeable when modifying with the lower tool rotational speed of 280 RPM (Fig. 2) and disappears with increasing the rotational speed (Fig. 3).

Arrangement of the material in the form of “onion rings” is visible, which is observed as a repeating pattern in the cross section of the weld. They arise as a result of a rhythmic change in the size and distribution of the strengthening phase. The patterns repeat at intervals (seen in the cross section) equal to the linear distance travelled by the tool during each rotation. The information presented in the literature indicates that it is related to oscillation of the tool rotation axis around its linear axis of motion [26, 27]. Higher rotational speeds generate more heat; in addition, in the stir zone, layers with different material temperatures should be expected [26, 28], which leads to the formation of bands richer in precipitates (layers with a higher temperature) and bands poorer in secondary phases (layers with a lower temperature) [27].

Figure 4 a and b show the microstructure of the studied alloy, while Figure 4c, d presents the micrographs of the microstructure showing the stir zone depending on the rotational speed of the tool used. The microstructure of the alloy before

Fig. 7 Microstructure of alloy after FSP treatment (450 RPM) with visible transition from FSP zone towards base material on advancing side and results of linear chemical composition analysis; SEM

Fig. 8 Microstructure of alloy after FSP treatment (450 RPM) with visible transition from area of lower FSP zone towards base material and results of linear chemical composition analysis; SEM

modification consists of a few rhomboidal/cubic precipitates with stoichiometry corresponding to the SnSb phase and numerous needle-shaped and globular-shaped precipitates with stoichiometry corresponding to the Cu_6Sn_5 phase distributed on the background of a tin-rich matrix (Fig. 4a, b), which is confirmed by the results of the EDS analysis (Fig. 5) and the X-ray phase analysis (Fig. 6). As a result of FSP modification, fine and equiaxed recrystallized grains of the tin-rich matrix (Fig. 4c, d) with a size of approx. 4–40 μm were formed in the microstructure. FSP generates a significant rise in temperature due to frictional forces, intense plastic deformation, and material flow caused by the movement of the tool, thus favoring dynamic recrystallization in the stir zone.

The EDS analysis of the chemical composition (Fig. 5) and XRD analysis of the phase composition (Fig. 6) showed no change in the phase composition of the alloy as a result of modification, while both the SnSb and Cu_6Sn_5 precipitates were significantly refined. The presented micrographs of the microstructure (Figs. 4 and 5) show a change in the morphology of the SnSb and Cu_6Sn_5 phases present in the alloy. The shape of the Cu_6Sn_5 phase particles changed to more regular, close to globular, while the SnSb phase occurs in the form of very numerous, small globular particles, evenly distributed in the matrix—inside the grains and at the grain boundary (Fig. 4c, d; Fig. 5 - point 1). Lead is evenly distributed in the tin matrix, and also occurs in the form of small precipitates with a

stoichiometry corresponding to the eutectic composition of Sn-Pb (Fig. 5 - point 4).

One of the advantages of the FSP method is the possibility of obtaining a microstructure that changes in a gradient manner from the surface into the depth of the product to prevent defects, e.g., spalling. The transition zone between the stir zone and the thermo-mechanically affected zone and the area directly under the pin are determined mainly by adhesion, which is very important in the case of surface engineering [12]. Linear analysis of the chemical composition was performed to accurately characterize the transition areas from the stir zone towards the base material. The results of the linear analysis are shown in Figures 7 and 8. Additionally, in Figures 7 and 8, the point corresponding to the clear transition boundary (point A) is marked with a dashed yellow line on the graph. The tests confirmed a significant increase in the refinement of the Cu_6Sn_5 and SnSb phase particles in the material stir zone after friction treatment on the advancing side (Fig. 7) and in the lower transition zone under the pin (Fig. 8). This is confirmed by the numerous peaks in the graphs corresponding to the composition of these phases.

In order to determine the influence of FSP on the microstructure of the studied alloy, including the process parameters, statistical analysis of the SnSb and Cu_6Sn_5 phases present in the microstructure was also performed. The results of the quantitative analysis including determination of the cross-sectional area of the

Fig. 9 Results of statistical analysis of Cu_6Sn_5 precipitates depending on FSP conditions. **a** Precipitate size histogram. **b** NND histogram; results are related to alloy after casting

Fig. 10 Results of statistical analysis of SnSb precipitates depending on FSP conditions. **a** Precipitate size histogram. **b** NND histogram; results are related to alloy after casting

particles and the nearest neighbor distance for the Cu_6Sn_5 phase are presented in Fig. 9, and for the SnSb phase particles in Fig. 10. The obtained results confirm the refinement of the microstructure as a result of FSP modification.

The particle size histogram of the Cu_6Sn_5 precipitates is characterized by an extremely asymmetric shape (Fig. 9a). A clear maximum in the histogram occurs for particle sizes under $25 \mu\text{m}^2$, which in the case of the initial material account for about 76% of the studied population. On the other hand, as a result of FSP modification, this range includes approx. 81–90% of the analyzed precipitates. The increase in rotational speed slightly influenced the refinement of the Cu_6Sn_5 phase. In addition, the results of NND measurements showed a shift in the population towards smaller distances between the precipitates for the material after FSP modification compared to the cast material (Fig. 9b). Moreover, the influence of rotational speed on the nearest neighbor distance was noticed. For the speed of 450 RPM, over 95% of the analyzed population is below $10 \mu\text{m}$, of which the nearest neighbor distance, less than $5 \mu\text{m}$, is shown by approx. 50% of the studied precipitates. For comparison, for the initial material, only 32% of the population was in this range.

The results of measuring the size of the SnSb phase precipitates presented in Fig. 10a confirm its considerable refinement after FSP modification. There was an almost tenfold increase in the percentage of the smallest particles with a size below $5 \mu\text{m}^2$ in the material after FSP treatment compared to the initial

material. The use of FSP modification practically eliminates precipitates larger than $20 \mu\text{m}^2$, while in the initial alloy, these particles account for nearly 78% of the studied population of precipitates. The nearest neighbor distance measurement results also confirm a significant increase in particle refinement as a result of FSP modification. The NND histogram (Fig. 10b) indicated a significant shift towards shorter distances. Virtually, the entire population of SnSb precipitates for the material after FSP modification is in the range below $15 \mu\text{m}$, including 40–50%—below $5 \mu\text{m}$, while in the case of the initial material, no particle was recorded in the smallest distance interval; in contrast, 91% of the studied particles are at a distance greater than $15 \mu\text{m}$.

3.2 Mechanical properties

The results of the Brinell hardness measurements for the material after FSP modification depending on the rotational speed of the tool are shown in Figure 11. Modification of the SnSbCu alloy by FSP caused a slight fall in hardness in relation to the initial alloy (20 HB), which is related to recrystallization of the alloy (Figs. 2, 3, and 4). No significant influence of the tool rotational speed on the hardness result was noticed; the obtained hardness values remain at the level of 17–18 HB. The modification using the higher rotational speed resulted in greater homogeneity of the microstructure, which is confirmed by microstructure observations (Figs. 2 and 3); therefore, there is a smaller measurement error when using a higher rotational speed.

The results obtained in the static compression test depending on the state of the SnSbCu alloy (as cast, after FSP treatment with different speeds) are shown in Fig. 12; the table summarizes the values of real stresses determined for all the considered variants of the alloy, which correspond to the actual strain amounting to 0.1, 0.2, and 0.3. The initial alloy is characterized by higher stress values in relation to the alloy after FSP modification, which can be explained by recrystallization of the tin-rich matrix. As a result of dynamic recrystallization of the material during FSP, the density of structural defects in the material is reduced. Therefore, there is a reduction in the strength properties of the material and improvement in its plastic properties.

Fig. 11 Comparison of SnSbCu alloy hardness before and after FSP modification

Fig. 12 Compression characteristics for SnSbCu alloy before modification and after FSP treatment with rotational speeds of 280 RPM and 450 RPM; table contains summary of stress values

Variant	σ , MPa at $\varepsilon = 0.1$	σ , MPa at $\varepsilon = 0.2$	σ , MPa at $\varepsilon = 0.3$
initial	117	149	190
280 RPM	100	137	165
450 RPM	100	126	157

The results of the flexural strength test (Fig. 13) showed its increase after FSP modification compared to the initial alloy, for which the flexural strength value was 214 MPa, and after the modification process, 307 MPa (280 RPM) and 290 MPa (450 RPM), respectively. The increase in flexural strength in the case of the alloy after FSP modification is caused by refinement of and a change in the morphology of the Cu_6Sn_5 phase particles to a more globular shape. It is worth noting that in the case of the samples after FSP treatment, cracks appeared on the advancing side, as shown in the photos of the samples after bending (Fig. 13). The results of the numerical simulations presented in [27, 28] indicate that the advancing side is usually the hotter side of the FSP zone due to the higher level of stress in relation to the retreating side in the modified material. This is related to the consistent direction of rotation and movement of the tool on

Fig. 13 Flexural strength of SnSbCu alloy before modification and after FSP treatment and photos of samples after bending—side view with visible fracture area

the advancing side, while on the retreating side, these directions are the opposite. In the case of a constant linear speed, with an increase in the rotational speed, the maximum temperature distribution shifts from the advancing side towards the retreating side [27]. On the other hand, the increase in the rotational speed of the Whorl tool contributed to the formation of a heterogeneous microstructure in the SZ of the B89 alloy, observed in the form of “onion rings” (especially visible on the advancing side), which may indicate turbulent material flow around the tool pin due to excess plasticized material under the shoulder with greater heat input. The research on this subject, presented in [27, 29], suggests that the formation of richer and poorer bands of precipitate is associated with uneven temperature distribution in the FSP zone. The flow of material layers with different temperatures is responsible for the heterogeneous distribution of the precipitates. The formation of a heterogeneous microstructure in the form of “onion rings” may increase the susceptibility to cracking on the advancing side.

In the case of the initial material, after the casting process, the fracture has a transcrystalline cleavable character within the Cu_6Sn_5 phases present in the microstructure. These phases are abundant in the microstructure, which was confirmed by microstructural studies. Additionally, in the area of the tin-rich matrix, the fracture has a transcrystalline ductile character; it is characterized by significant unevenness of the interface (Fig. 14a). After FSP modification, the fractures are of a different nature (Fig. 14b, c). There is an increase in participation of a transcrystalline ductile nature in the fracture, especially in the case of the material modified at the lower speed of 280 RPM (Fig. 14b). It is characterized by numerous dimples, indicative of plastic deformation during bending of the tin-rich matrix. Within the hard Cu_6Sn_5 phases, the fracture has a transcrystalline cleavable character. In the cast material, due to the morphology, the hard acicular Cu_6Sn_5 phases break much more easily than the fine, more

Fig. 14 Fractures after three-point flexural test. **a** Initial material. **b, c** After FSP modification; SEM

regular, globular-shaped ones occurring in the microstructure modified by FSP.

The nature of the fractures is consistent with the results obtained in the three-point flexural test; the material in its initial state after the casting process was characterized by the lowest bending strength, while after modification with the speed of 280 RPM, it was the highest.

4 Conclusions

Based on the research, the following conclusions were drawn:

- The use of FSP modification to treat the surface of the SnSbCu-bearing alloy causes a change in the morphology of the hard phases, including their strong refinement—confirmed by a rise in the share of the finest CuSn particles with an area under $25 \mu\text{m}^2$ and SnSb with an area under $5 \mu\text{m}^2$. In addition, FSP modification with the higher rotational speed of the tool results in greater refinement of the microstructure and its homogenization.
- The hardness tests revealed slight differences in hardness for individual sample variants. A slight change in the Brinell hardness of the studied material, as a result of FSP modification, is related to recrystallization in the stir zone, which results in a fall in Brinell hardness by approx. 10% compared to the cast material.
- The use of FSP treatment results in a reduction in the compressive stress. Additionally, it improves the flexural

strength, which can be explained by the increase in the proportion of fine particles of the CuSn and SnSb intermetallic phases and the change in their morphology.

Code availability Not applicable.

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Data availability The authors confirm that the data supporting the findings of this study are available within the article [and/or] its supplementary materials.

Declarations

Conflict of interest The authors declare no competing interests.

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