

Structural Behavior, Microstructure Evolution and Electrical Properties of $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ Synthesized by Thermal Decomposition Method under Different Sintering Temperatures

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ABSTRACT

This study investigates the influence of sintering temperature (900–1400 °C) on the structural, microstructural, and electrical properties of bulk $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ (PSMO) synthesized via a thermal decomposition method under ambient conditions. X-ray diffraction (XRD) analysis confirmed the formation of orthorhombic PSMO with a perovskite structure and space group Pnma (62) for all samples, while secondary PrMn_2O_5 phases were detected in samples sintered at 900 °C and 1000 °C. Single-phase PSMO was successfully achieved at sintering temperatures of 1100 °C and above. Scanning electron microscopy (SEM) revealed progressive grain growth and enhanced intergranular connectivity with increasing sintering temperature. Temperature-dependent resistivity measurements showed that the metal–insulator transition temperature (T_{MI}) increased from 230 K for the sample sintered at 900 °C to 240 K, 266 K, 268 K, 274 K, and 276 K for samples sintered at 1000, 1100, 1200, 1300, and 1400 °C, respectively, accompanied by a substantial reduction in resistivity. The highest resistivity of $1.60 \, \Omega \cdot \text{cm}$ was observed for the sample sintered at 900 °C, whereas the lowest peak resistivity of $2.59 \times 10^{-3} \, \Omega \cdot \text{cm}$ was obtained for the sample sintered at 1400 °C. The improved electrical performance at higher sintering temperatures is attributed to enhanced crystallinity, increased grain size, improved grain connectivity, and reduced electron scattering at grain boundaries. Overall, the sample sintered at 1400 °C exhibited the most favorable combination of phase purity, microstructural development, and electrical transport properties, indicating that this temperature provides the optimum processing condition for PSMO synthesized via thermal decomposition.

Keywords: Metal-insulator transition temperature; structural; microstructural; inter-grain connectivity; electrical resistivity

INTRODUCTION

Praseodymium strontium manganite with the nominal composition $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ (PSMO), is a member of the hole doped perovskite manganite family and exhibits notable electrical [1] and magnetic behavior resulting from the interplay among charge carriers, spin ordering, lattice distortions, and orbital interactions [2,3]. This compound typically undergoes a metal to insulator transition and displays pronounced magnetoresistive responses, which make it attractive for use in magnetic sensing devices, spin based electronic components, and magnetocaloric cooling technologies [4,5]. In addition to these conventional functionalities, praseodymium-based manganites have recently been incorporated into multifunctional composite systems for energy related applications, including hydrogen storage, highlighting their growing technological significance beyond purely magnetic devices [6].

Recent investigations on polycrystalline PSMO have reported pronounced electrical transport behavior together with a distinct metal-insulator transition, the characteristics of which are closely linked to structural features and microstructural development [7]. It has also been demonstrated that the structural, magnetic, and charge transport responses of PSMO vary considerably with the preparation route, as noticeable differences are observed among materials synthesized by solid-state reaction, sol-gel processing, and chemical co-precipitation [5,6]. A variety of preparation approaches, including conventional solid-state reaction [8,9], sol-gel techniques, and co-precipitation methods, have therefore been employed to produce PSMO and related manganites, often resulting in differences in phase formation, grain morphology, and electrical transport behavior depending on the chosen route [10]. Besides the synthesis route, sintering temperature is another critical factor governing the structural and electrical performance of manganites. Variations in sintering temperature can influence phase formation, grain growth, crystallinity, grain boundary density, and oxygen stoichiometry, all of which directly affect charge transport and metal-insulator transition behavior. Solid-state processing generally requires elevated temperatures and repeated grinding steps to obtain chemically homogeneous products [5,11], while wet chemical routes such as thermal treatment-based synthesis, sol-gel processing, and co-precipitation can improve compositional uniformity and reduce reaction temperatures [5,12-14]. However, these methods often involve prolonged processing times and the use of costly precursors, organic solvents, or chelating agents. Such differences in processing conditions strongly influence grain boundary connectivity and defect formation, which in turn affect the functional properties of manganites [15,16]. Despite extensive studies on synthesis routes, systematic investigations on the influence of sintering temperature on the structural, microstructural, and electrical characteristics of PSMO prepared using a simple and cost-effective synthesis method under ambient conditions remain limited.

Given these limitations, there is a clear need for preparation routes that are simpler to implement and more economical for producing PSMO with controlled microstructural features suitable for practical use. The thermal decomposition approach provides a convenient option because it employs readily available precursor compounds and involves comparatively uncomplicated processing steps. This method can shorten overall

processing time and reduce production costs, while still allowing regulation of phase development and particle growth [17-19]. In thermal decomposition process, precursor compounds are transformed into simpler oxide phases through heating, without the involvement of additional chemical reagents or solvents, as the applied thermal energy alone is sufficient to break chemical bonds [20]. The process is endothermic in nature and therefore requires continuous external heat input. Owing to its simplicity and versatility, thermal decomposition has been widely adopted in materials research, particularly for the preparation of metal oxides and ceramic powders [21-24]. For perovskite manganites, this route offers a practical and comparatively low-impact environmental alternative that enables improved phase formation and control over particle size, making it attractive for scalable material production.

In the present work, PSMO bulk samples were synthesized via a thermal decomposition method without a catalyst under environmentally friendly processing conditions. The effects of different sintering temperatures on the crystal structure, microstructure evolution, and electrical properties were systematically investigated. The aim is to elucidate the correlation between sintering temperature and the functional properties of PSMO to enable performance optimization for practical and large-scale industrial applications.

MATERIALS AND METHOD

Sample Preparation

In this study, $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ was synthesized using the thermal decomposition method. This approach was adopted based on the procedure previously reported in [21]. High purity metal acetates with approximately 99.9 % purity, namely praseodymium acetate hydrate $\text{Pr}(\text{CH}_3\text{COO})_3 \cdot x\text{H}_2\text{O}$, strontium acetate $\text{Sr}(\text{CH}_3\text{COO})_2$, and manganese acetate $\text{Mn}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ obtained from Alfa Aesar, were weighed according to the required stoichiometric molar ratios. The precursor powders were thoroughly mixed and ground for 1 h to ensure homogeneity. The mixed powders were calcined in air at 900 °C for 12 h. After calcination, the powders were reground for 30 min to improve uniformity. The finely ground powders were then pressed into pellets and sintered at different temperatures, namely 900, 1000, 1100, 1200, 1300 and 1400 °C (PSTD 900, PSTD 1000, PSTD 1100, PSTD1200, PSTD 1300 and PSTD 1400) for 24 h under ambient conditions using a tube furnace.

Sample Investigation

Phase identification and crystal structure analysis were performed using X-ray diffraction (XRD) with a PW 3040/60 MPD X'Pert Pro diffractometer (PANalytical Philips DY 1861). Cu- K_α radiation with a wavelength of 1.5406 Å was used. Diffraction patterns were recorded over a 2θ range of 20°- 80° with a step size of 0.03°. Rietveld refinement of the XRD data was carried out using HighScore Plus software. The microstructure and surface morphology of the samples were examined using scanning electron microscopy (SEM) (Phenom ProX G6, Thermo Fisher Scientific). Grain size analysis was performed using ImageJ software based on SEM micrographs. A total of 100 grains were randomly selected and measured to determine the average grain size distribution. The temperature-

dependent electrical resistivity of the samples was measured using a standard direct current (DC) four-point probe technique under cryogenic conditions with a helium gas atmosphere. A constant current was supplied through probes 1 and 4 using a Keithley 6221 DC/AC current source (USA), with an accuracy of $\pm 0.001 \mu\text{A}$, while the resulting voltage drop between probes 2 and 3 was recorded using a Keithley 2182 nanovoltmeter (USA), with a voltage resolution of $\pm 0.0001 \text{ V}$. Temperature control was carried out using a Lake Shore Cryotronics Model 335 temperature controller (USA), which provides a temperature stability of $\pm 0.001 \text{ K}$. The system was integrated into a closed-cycle cryostat capable of achieving low-temperature measurements down to liquid helium conditions without the need for external cryogenics. All measurements were performed over a temperature range of 30–300 K under vacuum and helium atmosphere conditions. The sample pellets were circular in shape with a diameter of $\sim 13 \text{ mm}$, and a probe spacing of 2 mm was used for all measurements. The measurements were carried out to determine the metal–insulator transition behavior of the samples.

RESULTS AND DISCUSSION

Structural and Phase Formation Analysis

The XRD patterns of PSMO samples sintered at various temperatures are presented in Figure 1, while the refined structural parameters derived from Rietveld analysis are summarized in Table 1.

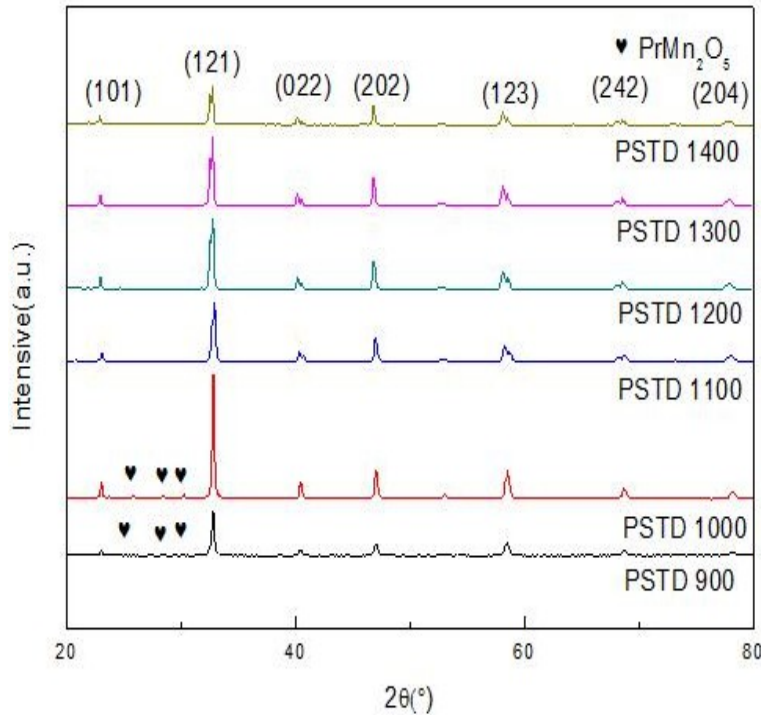


Figure 1. XRD patterns of PSMO samples sintered at different temperatures

All samples crystallized in the orthorhombic phase with space group $Pnma$ (62). Minor amounts of a secondary PrMn_2O_5 phase were detected in samples sintered at 900°C

(PSTD 900) and 1000 °C (PSTD 1000), with weight fractions of 7.4% and 5.1%, respectively, indicating incomplete phase formation at lower sintering temperatures. The refinement reliability factors (R_{wp} , R_p) and goodness-of-fit values (χ^2) confirm a satisfactory agreement between experimental and calculated diffraction patterns, indicating reliable structural refinement. The lattice parameters (a , b , c), together with Mn–O bond lengths and Mn–O–Mn bond angles, show only slight variations with increasing sintering temperature, suggesting that the fundamental crystal framework remains stable throughout the sintering process [25]. The crystallite size exhibits a general increasing trend from 900 °C to 1300 °C, reaching a maximum value of 83.6 nm at 1300 °C. However, a decrease in crystallite size to 60.9 nm is observed at 1400 °C. Such non-monotonic behavior has been reported in manganite systems and may be associated with enhanced grain coalescence and temperature-induced structural distortion during high sintering temperatures [26-28]. The reduction in FWHM values with increasing sintering temperature up to 1300 °C indicates improved crystallinity and enhanced structural ordering, supporting better phase development in the PSMO system [29].

Table 1. Rietveld refinement data of PSMO samples sintered at different temperatures

Sample code	PSTD 900	PSTD 1000	PSTD 1100	PSTD 1200	PSTD 1300	PSTD 1400
Phase	Praseodymium Strontium Manganese Oxide ($\text{Pr}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$)					
Reference code	98-002-9576					
Crystal system	Orthorhombic					
Space group	Pnma (62)					
a (Å)	5.456	5.457	5.460	5.459	5.460	5.457
b (Å)	7.713	7.715	7.720	7.717	7.718	7.715
c (Å)	5.485	5.483	5.484	5.483	5.483	5.484
V (Å ³)	230.838	230.844	231.133	231.013	231.044	230.863
Mn ₁ -O ₁ (Å)	1.959	1.959	1.960	1.948	1.960	1.959
Mn ₁ -O ₁ (Å)	1.963	1.962	1.963	1.969	1.963	1.963
$\angle\text{Mn}_1\text{-O}_1\text{-Mn}_1$ (°)	161.010	161.005	161.002	161.982	161.004	161.006
R_{EXP} (%)	4.219	3.337	3.946	4.209	4.024	4.655
R_p (%)	4.471	3.262	3.774	4.891	4.248	4.988
R_{wp} (%)	5.6970	4.2889	4.7666	6.5205	5.4403	6.6252
Goodness of fit (χ^2)	1.823	1.652	1.459	2.399	1.828	2.025
FWHM values (°)	0.216	0.108	0.130	0.159	0.113	0.161
Crystallite size (nm)	43.8	57.5	66.2	77.4	83.6	60.9
Secondary phase	Praseodymium Mangano(III)manganate(IV) (PrMn_2O_5)					
Reference code	98-007-6124					
Crystal system	Orthorhombic					
Space group	Pbam (55)					
a (Å)	7.556	7.549	-	-	-	-
b (Å)	8.639	8.646	-	-	-	-

c (Å)	5.707	5.708	-	-	-	-
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Microstructural Analysis

Figure 2 illustrates SEM images of all PSMO samples. Grain size histograms were obtained only for PSTD 900 and PSTD 1000 (Figure 3), where individual grain boundaries could be clearly distinguished, allowing reliable grain size measurements. The average grain size increased from approximately 200.98 nm for PSTD 900 to 284.03 nm for PSTD 1000, indicating that higher sintering temperatures promoted grain growth and an increase in crystallite size (Table 1) through enhanced particle coalescence [10, 30]. For samples sintered above 1000 °C, extensive grain coalescence and increasingly indistinct grain boundaries prevented accurate grain size determination. At the lowest sintering temperature of 900 °C (PSTD 900), the microstructure consists of relatively small, inhomogeneous, and agglomerated grains with noticeable porosity. With increasing sintering temperatures above 1000 °C, grain boundaries become less distinct due to enhanced diffusion and stronger particle coalescence, resulting in improved intergranular connectivity. At 1100 °C (PSTD 1100), the microstructure exhibits strongly fused grains accompanied by porosity and poorly defined grain boundaries. As the sintering temperature increases further to 1200 °C and above, neighbouring grains merge through necking growth at grain boundaries. The continued coalescence and grain-boundary diffusion produce larger consolidated particles with indistinct grain boundaries and melt-like microstructural features, as observed in samples sintered at 1200 °C and above [31].

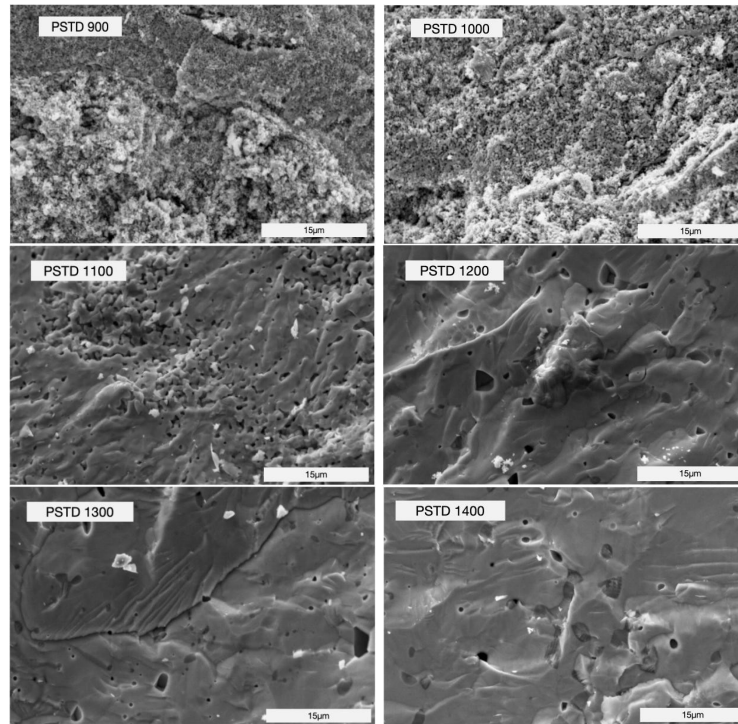


Figure 2. SEM micrographs of the PSMO surface at different sintering temperatures

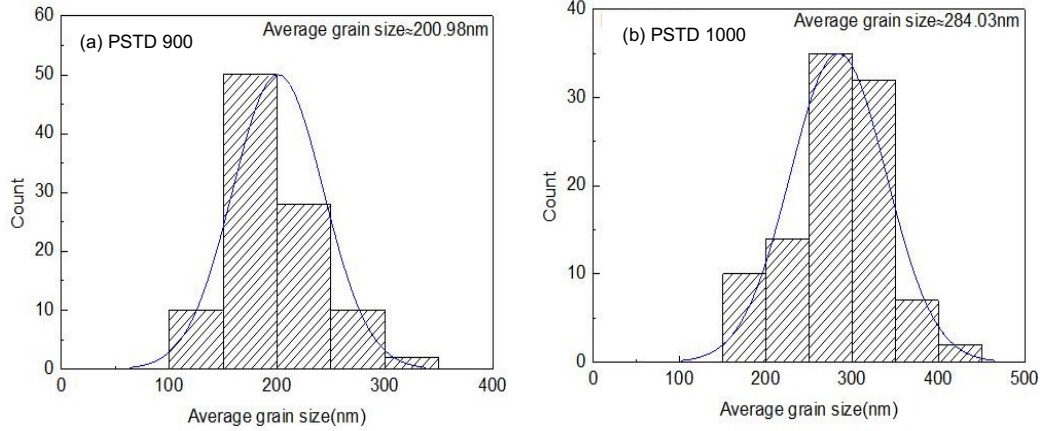


Figure 3. Grain size distribution histograms of PSMO samples sintered at (a) 900 °C (PSTD 900) and (b) 1000 °C (PSTD 1000)

Resistivity Measurement Analysis

Figure 4(a) presents the temperature-dependent resistivity ($\rho-T$) curves of PSMO samples sintered at different temperatures, while Figure 4(b) provides an enlarged view of samples sintered at 1100 °C, 1200 °C, 1300 °C, and 1400 °C. The metal-insulator transition temperature (T_{MI}) was determined from resistivity measurements performed over the temperature range of 30–300 K using the four-probe technique. As shown in Table 2, the T_{MI} values increase systematically with sintering temperature, from 230 K for PSTD 900 to 240 K for PSTD 1000, 266 K for PSTD 1100, 268 K for PSTD 1200, 274 K for PSTD 1300, and 276 K for PSTD 1400. The sample sintered at 900 °C (PSTD 900) exhibits the highest resistivity and the lowest T_{MI} , which can be attributed to enhanced charge carriers scattering at grain boundaries arising from magnetic disorder and poor connectivity between small grains [32]. As the sintering temperature increases, the progressive increase in T_{MI} and reduction in resistivity indicate improved grain connectivity and crystallinity, thereby facilitating the charge transport process [33–35]. The most significant increase in T_{MI} occurs between PSTD 1000 and PSTD 1100, consistent with the significant microstructural evolution observed in the SEM images. These observations demonstrate a clear correlation between microstructure and electrical transport properties. Enhanced grain growth and particle coalescence at higher sintering temperatures improve inter-grain connectivity and reduce electron scattering at grain boundaries, thus lowering resistivity and enhancing the overall electrical conductivity of the PSMO samples. For comparison, the T_{MI} values obtained in this work are comparable to those previously reported for PSMO manganites synthesized via conventional solid-state and sol-gel routes [3,15], which typically exhibit T_{MI} values in the range of ~220–280 K depending on grain size and oxygen stoichiometry [5,36]. This indicates that the present samples synthesized using thermal decomposition method possess electrical transport properties comparable to those reported in the literature.

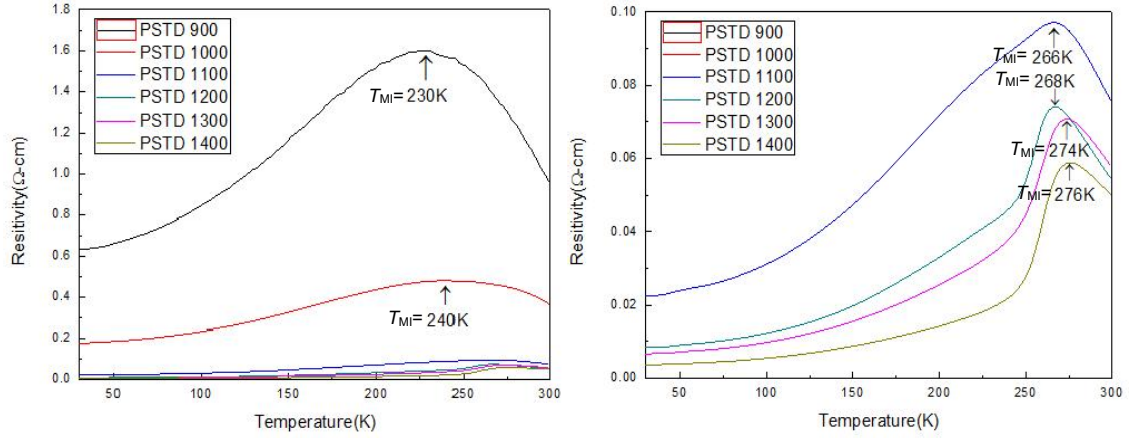


Figure 4. Electrical resistivity as a function of temperature for (a) PSMO samples sintered at different temperatures, and (b) an enlarged view of PSMO samples sintered at 1100 °C, 1200 °C, 1300 °C, and 1400 °C

Table 2. Metal-insulator transition temperature (T_{MI}) of PSMO samples sintered at different temperatures

Sample Code	T_{MI} (K)
PSTD 900	230
PSTD 1000	240
PSTD 1100	266
PSTD 1200	268
PSTD 1300	274
PSTD 1400	276

CONCLUSIONS

In this work, the influence of different sintering temperatures on the structural, microstructural, and electrical properties of PSMO samples was systematically investigated. XRD analysis confirmed that a pure PSMO phase was achieved only at sintering temperatures of 1100 °C and above, whereas lower sintering temperatures (900 °C and 1000 °C) led to the formation of secondary phase (PrMn_2O_5), which adversely affected the microstructure and electrical behavior. With increasing sintering temperature, grain growth and crystallinity generally improved, and secondary phase were largely absent. Temperature-dependent resistivity measurements showed that the sample sintered at 900 °C exhibited the lowest T_{MI} of 230 K with the highest resistivity of $1.60 \, \Omega \cdot \text{cm}$. In contrast, the lowest peak resistivity of $2.59 \times 10^{-3} \, \Omega \cdot \text{cm}$ was observed for the sample sintered at 1400 °C, indicating enhanced charge transport resulting from improved grain connectivity and reduced electron scattering at grain boundaries. This behavior suggests that PSMO undergoes progressive agglomeration and thermally induced grain coalescence at higher sintering temperatures, leading to microstructural densification, defect evolution, and corresponding changes in electrical transport

properties. The thermal decomposition approach employed in this study not only reduces costs compared to other dry chemical synthesis methods but also aligns with environmentally friendly practices, providing a green route for producing PSMO powders.

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AUTHORS' CONTRIBUTIONS STATEMENT

The manuscript was written through the contributions of all authors. K.P.L, L.K, X.T.H, N.R and L.N.L designed and organized this work. S.H.Y, K.P.L, and L.K, X.T.H. writing-review and editing. X.T.H, K.P.L and N.R draft the main ideas of the work. K.P.L, M.M.A.K, S.K.C, M.K.S and A.H.S supervising. S.H.Y, K.P.L, L.K., X.T.H and L.N.L review and editing of the manuscript. All authors have approved the final version of the manuscript.

CONFLICT OF INTEREST

All authors declare no competing interests that could have influenced the work reported in this paper.

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