

# Superplastic Deformation Processes of the Mg Alloys: A Detailed Review

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## Abstract

The superplasticity of magnesium alloys (Mg) exhibits excellent behavior at high elongations while maintaining mechanical properties and a deformation mechanism that depends on intergranular sliding. These alloys can be achieved at lower stress levels with relatively high temperatures. Furthermore, the microstructure is fine-grained and homogeneous, which gives the alloy a very high potential for forming then, obtaining complex components of various weights. The microstructure and mechanical properties make magnesium alloys a successful choice for many industrial applications, including the automotive, aviation, and medical device industries, which require lightweight materials with high strength. The superplastic behavior of magnesium alloys treated by various manufacturing operations, such as differential speed rolling (DSR), equal channel angular pressing (ECAP), and friction stir processing (FSP) was studied. These operations contribute to reducing grain size and enhancing intergranular sliding, which is the basic mechanism responsible for superplasticity.

**Keywords:** Superplastic, Magnesium alloys, Differential rolling speed (DSR), Equal channel angular pressing (ECAP), Friction stir processing (FSP)

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## 1. Introduction

Superplastic is a polycrystalline material that exhibits large tensile elongations exceeding 400% and can withstand large deformations before failure [1]. Many techniques, such as symmetric rolling and electroslag remelting (ESR), are used to improve and develop metal alloys during grain refinement [2, 3]. There are requirements for the conditions of superplastic behavior of alloys in general, which include the deformation temperature higher than 0.5  $T_m$ , tensile strain rate 0.00001 to 0.1  $s^{-1}$ , granule size smaller than 10  $\mu m$ , and flow stress <10 MPa [4]. The most important contribution of superplastic deformation is grain boundary slip (GBS), which involves grain slip at the interface under an externally applied force [5-6]. Grain phase size (GPS) and severe plastic deformation (SPD) are employed to achieve a grain size of lower than 10  $\mu m$  with an equiaxed structure, thereby addressing the issue of low formability at high temperatures.

This approach also reduces manufacturing costs and time. The new material, subjected to superplastic conditions, can be used to manufacture complex products with high elongation and without premature failure. Two types of superplasticity are unique to the field of materials science from the perspective of using superplastic forming

methods for commercial applications, which are less temperature superplasticity and more strain rate superplasticity [26]. The superplastic forming (SPF) techniques, including hollow cavity, rapid plastic forming, blow molding, and diffusion bonding, can be applied to obtain complex and lightweight components with high dimensional accuracy [7]. The advantage of SPF can manufacture complex parts on small-sized equipment. According to the analysis of literature, it was found that most grain improvement methods depended on plastic deformation techniques, including FSP, HTP, DSR, and ECAP [8-16].

Magnesium alloys are usually obtained in fine and ultrafine grains by methods of severe plastic deformation, and they are prepared with elongation exceeding 400%. Magnesium alloys have excellent mechanical features at ambient temperature, a high specific strength, and ductility at moderate temperatures, as well as light weight. [17, 18]. The magnesium alloys lack anisotropic elements and have poor sliding properties at room temperature, with unsatisfactory ductility [19, 20]. Although the formability of magnesium improves at ambient temperature, its premature fracture rate limits its industrial use. The superplastic materials are widely used in automobiles (e.g.,

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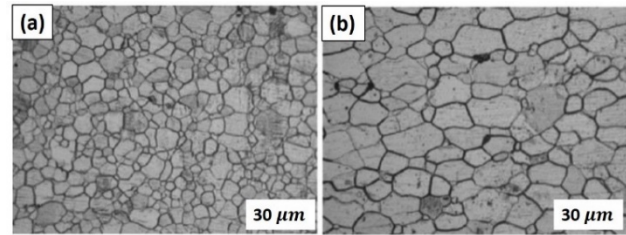
Toyota, Ford, Volkswagen, and General Motors) and in the world-renowned aviation industry (e.g., Embraer, Airbus, and Boeing), as well as in other applications [21-22]. On the other hand, they are applied in the manufacture of mobile phone covers, cameras, laptops, and computers, as well as in the field of electronics and protective devices. Magnesium features are low density, ease of manufacturing, electromagnetic protection, possibility of recycling, better heat dissipation, characterized by its good vibration damping and high strength-to-mass ratio [23]. It is known worldwide that its formation is weak at room temperature due to its complex deformation behavior, which is attributed to its hexagonal close-packed (HCP) lattice structure that is tightly packed, and the limited number of sliding systems [24, 25].

## 2. Superplastic Materials Process

According to the previous studies and research on superplastic materials, many works have been conducted using a variety of alloys. In this article, various types of Mg alloys and different methods are employed to investigate the plastic deformation properties under different temperatures and strain rates, aiming to enhance the microstructure, achieve high elongation, improve formability, and expand the applications of magnesium alloys. AZ31, AZ61, TZ61, TXM210, Zw13, and AZ41 magnesium alloys are adopted. The differential speed rolling is considered the most important process utilized to obtain superplastic behavior of magnesium alloys. Therefore, most researchers have addressed the issue of differential rolling speed to improve elongation and superplasticity, modify the crystalline texture to reduce slip, and also to achieve smaller and finer grain sizes compared to conventional rolling and other methods. The important rolling parameters, such as the velocity ratio, reduction rate per pass, and processing temperature, can affect the microstructure and mechanical properties.

H.G. Jeong et al. [27] used a differential rolling process (DRP) to form AZ31 alloy at a high-speed ratio of 3. This process was performed on a sample with an initial temperature of 473 K°. The thickness of the sheet was 2 mm, which was rolled to different reductions of 30%, 50%, and 70%. The high-angle grain boundaries (HAGBs) were recorded to be 0.53, 0.41, and 0.47 at the reduction of 30%, 50%, and 70%, respectively. The results of the microstructure based on the grain size measurement after grip and tensile elongation are shown in Fig. 1. After the 50% and 70% reductions, low-angle grain boundaries (LAGBs) in terms of sub-grains formed due to the increased overlap density, which then transformed into HAGBs through dynamic recrystallization [28]. The elongation increased with decreasing strain rates to 830% at  $(2 \times 10^{-4})/s$ , a temperature of 673 K°. DSR specimens showed significantly greater elongation than conventional rolling at 4.5  $\mu m$ . As a result, reducing the grain size increases the superplasticity [29]. The sliding of the grain boundary occurs at lower rates, and grain boundary climbing occurs at higher rates, while at 673 K°, the grain boundary sliding mechanism is completely dominant [30]. At 673 K° and  $(2 \times 10^{-4})/s$ , an elongation of 830% was

achieved, and the dominant mechanism was GBS with dynamic grain growth from 24 to 34  $\mu m$  instead of DRX, consistent with the expected critical size ( $\approx 29 \mu m$ ) and thus grain growth during tension.



**Fig. 1** Microstructure in areas (a) grip (b) measured after tensile elongation [27]

Dan Lu et al. [31] used the cast-roll method to manufacture magnesium alloy AZ31 at different speeds. The results showed that DSR weakens the basal texture, resulting in improved mechanical properties. At speed 1.2, the best results of grain size were obtained (10.3  $\mu m$ ), with the tensile stress of 240 MPa, yield stress of 145 MPa, and elongation of 28.5% at room temperature. It was found that rolling by the DSR method a significant enhancement in the elongation property compared to identical rolling, due to the poor basal crystallographic texture, with probable activation of non-basal slips and grain boundary sliding (GBS). S. Taylor et al. [32] studied a magnesium alloy (AZ41) employing a hot-rolled at high-temperature tensile testing method with (350–450°C) and strain rates (0.1 - 0.001  $s^{-1}$ ) to determine the deformation before failure. An Aramis GOM camera and a circular grid technique were adopted to simulate complex shapes, and the microstructure was studied using SEM, EBSD, and TEM. The high of the elongation reaches 520% under the optimum conditions of 450°C and a strain rate of 0.001  $s^{-1}$ , while under normal conditions, such as 400°C and a strain rate of 0.01  $s^{-1}$ , the elongation was 170%, 0.01  $s^{-1}$  at both 450 °C (195%), which is industrially acceptable. The main forming mechanisms are grain sliding at the edges and dynamic recrystallization (the internal structure changes during forming, i.e., regenerates), which means the alloy expands without breaking. In the gas swelling test, a 52 mm dome height was achieved in 12 minutes, which is the best performance for an aluminium alloy. It did not exhibit the premature melting seen in other alloys, due to the low amount of aluminium used in the alloy.

Watanabe, Hiroyuki, et al. [33] conducted research superplastic properties of ZK60 alloy at low temperatures, and the typical grain structure of 6.5  $\mu m$ . Performing tensile tests at low temperatures revealed a superplasticity of no more than 400%. A portion of the overall strain is due to grain boundary sliding (GBS), and the rate of strain is inversely related to both the cube of the grain size and the second stress force. The GBS-adapted dislocation motion controlled by grain boundary propagation is the deformation mechanism of LTSP.

Kim, W.J., et al [34] generated severe plastic deformation of AZ61 sheets by differential rolling at high speeds, resulting in grains with a fineness of 0.5-0.3  $\mu m$  in a single pass. Rolling temperature of 473-532 K° and a

thickness reduction of 70% showed good superplasticity, and the elongation reached 850% with a strain rate of  $0.003 \text{ s}^{-1}$ .

Kamil, Majchrowicz et al. [35] conducted an extended study on two newly developed magnesium alloys, AZ61 and TZ61, which were received and prepared as rectangular samples, heat-treated at  $480^\circ\text{C}$  for 1 hour, then rapidly cooled in water. After that, the samples were rolled based on DSR in four passes at a reduction rate of 15%, heated at  $400^\circ\text{C}$  for 10 minutes before each pass, and rotated  $180^\circ$  around the rolling direction [36]. Two rolling methods were applied: symmetric rolling at  $R=1$  and asymmetric rolling at  $R=3$  at a fixed roller speed of 4 m/s [37]. Asymmetric rolling proved effective in filtered grains, weakening the texture and developing the mechanical properties. These results indicate that there was a possibility for achieving superplasticity in TZ61 and AZ61 alloys after processing them by rolling at varying speeds, resulting in enhanced superplasticity due to a tiny grain size, reduced intensity of the primary crystal structure, and improved particle distribution.

Roberto B. Figueiredo et al. [38] used the ECAP process to enhance the processing of AZ31 magnesium alloy with a grain scale equal to  $2.2 \mu\text{m}$ . The annealing at  $673 \text{ K}^\circ$  for half an hour was performed. The alloy obtained excellent superplasticity at  $623$  and  $723 \text{ K}^\circ$ , with elongation reaching 1000% at strain rates less than  $0.0001 \text{ s}^{-1}$ . The experimental results were found to be consistent with the superplastic model for the intergranular sliding, and a strain rate sensitivity of 0.5.

Roberto B. et al. [39] employed the ECAP method to improve the grain structure for the superplastic properties of Mg alloy type ZK10. The grain refinement was obtained after four cycles of ECAP, and the elongations were improved at low strain rate with temperatures of  $473$  and  $523 \text{ K}^\circ$ . It was found that there was agreement between the experimental and theoretical models for the superplastic behavior. At high stress rates and temperatures, a transition to viscous sliding occurs.

Tomáš Vávra et al. [40] used equal-channel angular compression of the prepared commercial magnesium alloy WE43 to obtain a fine-grained structure with an average grain size of about  $340 \text{ nm}$  with a high secondary phase density. The possibility of lowering the temperature at strain rates of  $0.01 \text{ s}^{-1}$  and  $0.1 \text{ s}^{-1}$  compared to previous studies was considered. The negative effect of grain growth was controlled, and an elongation of 1230% at  $0.01 \text{ s}^{-1}$  at temperatures of  $350$  and  $400^\circ\text{C}$  was shown. Increasing the strain rate by 0.1 at  $400^\circ\text{C}$  resulted in 1000% elongation at maximum. The results showed that even after large deformation, the microstructure was fine-grained, and cavity growth occurred at  $450^\circ\text{C}$ .

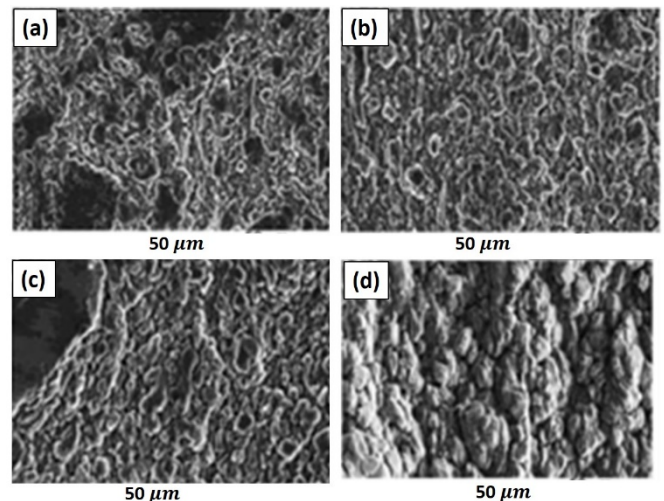
Marta Álvarez-Leal et al. [41] extruded a commercial magnesium alloy ZK30 at high temperatures to evaluate mechanical behavior through different temperatures and stress rates. The tensile behavior was associated with the microstructure and developed during testing to a  $30 \mu\text{m}$  with small grains ( $1\text{-}5 \mu\text{m}$ ). Elongations to failure

indicated 360% and the apparent stress exponent was low between 2.6 and 2.9 at lower stress rates. Stress exponent rates exceeding 2 are attributed to accelerated grain growth. The topography of the deformed sample at different temperatures is presented in Fig.2.

Zhao-Peng Yu et al. [42] select AZT910 magnesium alloy fine-grained  $5 \mu\text{m}$  after 16-cycle rolling to study the properties of tensile properties and the microstructure characteristics using three types of tensile specimens under room temperature, as well as  $200^\circ\text{C}$  and  $300^\circ\text{C}$ . The results revealed that tension direction properties correlate with room temperature and  $200^\circ\text{C}$ , while a negligible dependence at  $300^\circ\text{C}$ . This is due to basal and non-basal slips at room temperature, a mixture of basal and non-basal slips, and grain boundary rotation slips at  $200^\circ\text{C}$ . Grain boundary sliding (GBS) emerges as the predominant deformation process at  $300^\circ\text{C}$ .

Mengran Zhou et al. [43] utilized stir processing to obtain a superplastic behavior of magnesium alloy type Mg 9Li-1Zn, with a  $0.61 \mu\text{m}$  alpha particle size and a  $0.96 \mu\text{m}$  beta phase. The ductility was 369% 1104% at  $473\text{k}$  at a strain rate of  $0.1 \text{ s}^{-1}$  -  $0.0001 \text{ s}^{-1}$ .

Seyed Alireza Torbati Sarraf et al. [44] adopted a high-pressure torsion technique for up to 5 passes of ZK60 alloy to discover a grain size of  $9.4 \mu\text{m}$  as well as structural observations using an optical and scanning electron microscope. The results showed that superplastic behavior was achieved with a maximum elongation to failure of 535%, showing agreement with previous studies.



**Fig. 2** Topography of deformed sample at different temperatures: a. $300^\circ\text{C}$ , b. $350^\circ\text{C}$  c. $400^\circ\text{C}$ , d. $450^\circ\text{C}$  [41]

The hot rolling process was for the superplastic obtaining of the Magnesium alloy WE43. The impact of the strain rate on the elongation at  $440\text{-}500^\circ\text{C}$  was studied. The results revealed that the important superplasticity was performed at  $460^\circ\text{C}$  with a small grain size, weakest texture, and even distribution of phase number 2. The elongation magnitude reached  $367.7 \pm 3.7\%$  at a strain rate of 0.01. The observation of superplasticity at high strain rates indicates that the alloy deformation is controlled by grain boundary sliding and solute-dislocation creep [45].

Y.S. Kim et al. [46] applied the high ratio of the DSR technique on the microstructure of Mg9Al-1Zn (AZ91) alloy obtained by the casting procedure to enhance grain size and to show a semi-structured based on the eutectic  $\beta$ -phase. Optimum superelasticity was achieved at 573K° at 0.001 s<sup>-1</sup> with a maximum elongation of 830% due to the loss of thermal stability. As a result, DSR led to an important enhancement in the beta phase; the grain size was reduced from 53  $\mu\text{m}$  to 1-2  $\mu\text{m}$ .

W.J. Kim, et al. [47] employed fine-grained 1%Ca-AZ80 (1CaAZ80) magnesium alloys manufactured by a high differential speed rolling mill (DSRM). The particle size was decreased in the range 11.5 to 0.8  $\mu\text{m}$ , and the granules were purified and dispersed. At room temperature, it has a high yield strength of 340 MPa, and good superplasticity, with an elongation percentage of 700% determined at 523 K°.

Giuliano, Gillo et al. [48] selected AZ31 magnesium alloy to run the sensor strain modulus determined from free forming options at constant stress using a simplified analysis model. They adopted the strength-stress strain relationship when the strength modulus was determined by inverse analysis with FEM. The result showed that the simulation agreed well with the experimental analysis, with an error of less than 9% for predicting the forming time. This confirms the reliability of the model in describing the superplastic behavior of alloys.

Deepika Harwani et al. [49] performed high-speed two-pass friction stir (FSP) processing for the Mg-3Al1Zn alloy. Equiaxed fine grains of 6.15  $\mu\text{m}$  were obtained, which encouraged the development of large grains and the superplastic behavior of the alloy. Microstructure and tensile procedures were investigated at 350, 400, and 450°C, with a value of strain rate of  $1.3 \times 10^{-3}$  s<sup>-1</sup>. The flow stress reduced, and the elongation augmented at elevated temperatures. Superplasticity was observed, with an elongation of 200% at the highest deformation temperature of the specimen.

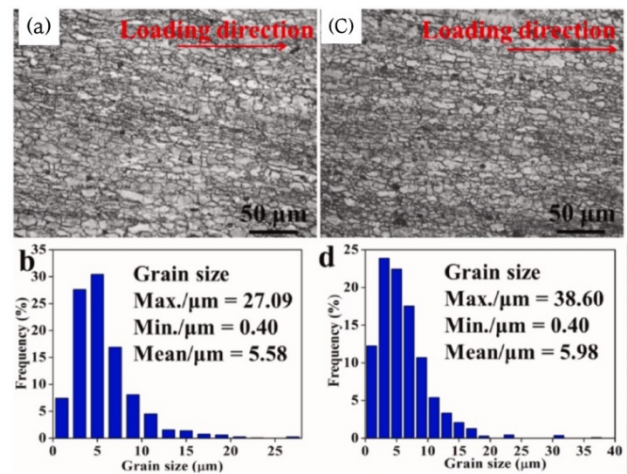
Marta Álvarez-Leal et al. [50] conducted friction stir processing (FSP) to evaluate ultra-fine grains with equal axes and to improve coarse deposits of WE54 alloy. The results showed high ductility of the material at ambient temperature and excellent super-strength at 0.01% strain, reaching an elongation of 726%.

Liqiang Zhan et al. [51] achieved hot extrusion at 400°C of the Mg-Nd-Zn-Zr alloy. The results revealed that the elongation was higher, 1015.50% at 450°C, to obtain fine grains. On the other hand, the ECAP technique was used with several passes at different temperatures. After 8 passes, the tensile property of the ZE41 magnesium was found to be higher, with a strain rate of 0.0001. At a strain rate of superplasticity, the grain boundary slip was 0.52-0.42 at 350 - 450 °C, and the solute drag creep was 0.37 at 500°C.

Taekyung Lee et al. [52] applied quickly solidified flaky powder metallurgy (RS FP/M) to determine superplasticity of AZ91 alloy at high strain rate, with elongation magnitude to failure of 465%. The best strain

rate to elongation was 0.01 s<sup>-1</sup>, which is higher than other methods. It resulted in a very effective improvement of ~1  $\mu\text{m}$  grain size and high thermal stability.

Abdul Malik et al. [53] conducted a study on the superplasticity of extruded ZK61 alloy based on the tensile test at elevated temperatures and multiple strain rates. The results showed that high flexural elongation between 400% and 334% at 673 K° and 623 K° with a strain rate of 0.001 s<sup>-1</sup>. The composition is thermally stable at high temperatures, the structure is also thermally stable, and intergranular sliding is a controlling deformation mechanism. All these factors helped to determine the superplasticity behavior of this alloy. Fig. 3 (a-c) presents that at a strain rate of 0.001 s<sup>-1</sup> under specific conditions, the microstructure developed. The results of OM depicted that the grains elongated at a stress rate of 0.001 s<sup>-1</sup> and a temperature of 623 K° developed within the sample, which is a sign of intra-grain sliding. Fig.3 (b-d) illustrates that during the tensile test and high temperature, the grain size developed and increased slightly. This increase is within the framework of superplasticity.



**Fig. 3** OM and SEM results for a strain rate of 0.001 s<sup>-1</sup> at the same temperature (a-b) 623k, and analyze it for grain size (c-d) at 673k and grain size analysis [53]

Alsubaie Saad et al. [54] applied high-pressure torsion (HPT) on AZ80 Mg alloys to improve the mechanical properties, increase plasticity, and the possibility of superplasticity at high temperatures. The results showed that superplasticity appeared with an elongation of 645% at 573 K°, and the presence of low temperature, while it was found at 473 K°, equal to 423%.

Edalati, K et al. [55] produced Mg-8 wt.%Li alloy ultrafine grains by severe plastic deformation based on an extrusion procedure. The results showed that the elongation reached 440% at room temperature, the strain rate sensitivity was 0.37, and the grain boundary sliding contributed about 60% of the total elongation.

Palacios-Trujillo, César, et al. [56] achieved superplasticity in ZK60 magnesium alloy through indirect extrusion at intermediate temperatures. The results revealed a homogeneous microstructure with a grain size of  $4 \pm 2$   $\mu\text{m}$ . The highest ductility was obtained at 250 °C

and  $10^{-4} \text{ s}^{-1}$ , with an elongation at failure of 464%. Grain Boundary Sliding – GBS is the dominant deformation mechanism.

Table 1. Reports on superplastic magnesium alloys under various conditions

Alloy	Processing Technique	Temperature (°C)	Strain Rate (s <sup>-1</sup> )	Grain Size (µm)	Elongation (%)	Reference
AZ31	DSR	200 °C	$1 \times 10^{-2}$ $2 \times 10^{-4}$ $2 \times 10^{-4}$	4.35	Low at 400 °C (673 K)	27
				3.7	Higher than 30% at 400 °C (673 K)	
				2.02	830% at 400 °C (673 K)	
AZ61	DSR	400 °C	—	~14.5	17–18%	35
				~10.8		
AZ31	ECAP	350–450°C	0.0001	6	1000%	38
ZA10	ECAP	250°C	0.0001	5.2	750%	39
WE43	ECAP	400°C	0.01	0.34	1230%	40
			0.1		1000%	
ZK30	Extruded	450°C	0.001	1-5,30	360%	41
AZT910	Extruded, Rolling	300°C	0.001	5	-	42
Mg-Li-Zn	FSP	200°C	0.0001	0.61-0.96	1104%	43
			0.01	0.61-0.96	396%	
ZK60	HPT	200°C	0.0001	1	535%	44

### 3. Conclusion

In this work, previous literature has been extensively reviewed based on the superplasticity of magnesium alloy, and many conclusions were summarized:

- 1- Magnesium alloys have good formability at superplasticity and high elongation without fracture. Intergranular sliding of GBS plays an important role in superplasticity formation at elevated temperature and low strain rate.
- 2- Method of differential speed rolling DSR shows that with increasing thickness reduction, grain refinement increases, tensile elongation increases with decreasing strain rate. On the other hand, the stress hardening rises with decreasing strain rate and with increasing temperature.
- 3- It was shown that the difference between the elongation and damage of AZ31 alloy sheets treated with DSR and ESR technology decreased at high temperatures, which led to a weakening of the basal tissue in elongation at high temperatures
- 4- In the ECAP technique, the possibility of achieving superplastic elongations at the lowest strain rates was demonstrated. It was also shown that the true stress curves in the direction of the investigated stress

revealed clear hardening and high sensitivity in the strain rate, which is compatible with large elongations.

- 5- In the extrusion process, it was found that there was an improvement in the superplastic behavior with elongation, as well as clear evidence of the presence of GBS and the microstructure developed after extrusion.
- 6- Ultra-fine grains with equal axes, high ductility of the material at ambient temperature, and excellent super-strength at 0.01% strain, reaching an elongation of 726% of WE54 alloy, can be obtained using friction stir processing (FSP).

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### Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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