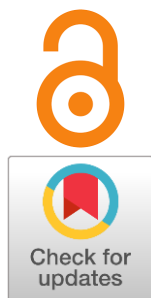


The present work is focused on the analysis of the process of fast sintering of a powder matrix at low temperatures, when the atomic diffusion rate is low. A resonant sintering model based on phonon vibration of particles is proposed. The analysis of the conditions necessary for the implementation of the process of fast sintering of ceramics is presented.



keywords: ceramics, fast sintering, resonant sintering, dilatometry

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1. Introduction

Ceramic materials have been known since ancient times and have been widely used in all areas of human activity. In the ceramic technology, we understand the production of the polycrystalline inorganic materials by powder molding and sintering. This method is referred to as “powder metallurgy” and it is used to obtain compact metals with high melting temperature.

Despite seeming simplicity of the method, the formation of a dense ceramic material, including solid oxide electrolytes, is not a simple task. Dense ceramics does not have open porosity and its density should exceed 95 %. Ceramics sintering is a complex process that is sensitive to many parameters. Therefore, depending on the great variety of parameters, the sintering does not proceed according to a common mechanism.

Reduction of sintering temperatures is a trend of modern methods of different types of ceramics. High temperatures are not acceptable for fine crystalline ceramics; such conditions are not technologically beneficial and costly for industrial processes. Moreover, high sintering temperatures do not always solve all problems. For example, an increase in the temperature threshold does not always result in the higher material density, but also causes its decrease due to the dissociation of evaporation processes.

The monograph by professor Rahaman [1] describes in detail the methods, processes, and theory of the dense ceramics formation. The monograph illustrates different models of the processes that take place during sintering of ceramic materials. These models are based on the atomic diffusion and correlate well with the results of the high temperature experiment. However, the basic and at the same time the fastest sintering of the powder matrix proceeds at relatively low temperatures, when the atomic diffusion is low. This is a promising temperature region for dense fine crystalline ceramic formation at low temperatures.

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The monograph [1] inspired me to express my humble understanding of the fast sintering process and conditions for the dense ceramics formation. My more than 50-year research work has been devoted to the synthesis and study of various types of ceramic materials, mainly solid electrolytes. The presented paper has few references, as the author shares with the reader the results of a half century experience and thoughts that have not been previously published. The references of the monograph [1] and vast information on ceramics reported in it provide significant amount of literature data as well as correlation with the experimental data. The paper presents some technological issues, which are vital for the sintering process.

2. On the physics of ceramics fast sintering

The development of a method for the production of desired dense ceramics by the variation of the synthesis conditions is a difficult and time-consuming research. The method of dilatometry allows determining the main peculiarities of the powder matrix sintering and suggests the possible reasons influencing this process.

The classical dilatometry sintering curve has a form reminding of an elongated phase transition (Figure 1). Basically, this appears to be the case, if we consider the transformation of the surface structure during the sintering to the volume as a phase transition. This form of the curve has a monoclinic tetragonal transition in ZrO_2 [2]. Dilatometric sintering curves of this type are provided in [3] for three solid electrolytes: $Ce_{0.9}Gd_{0.1}O_{1.95}$; $0.92ZrO_2 + 0.08Y_2O_3$ and $0.89ZrO_2 + 0.10Sc_2O_3 + 0.01CeO_2$.

The sintering process, as illustrated by the dilatometric curve, has three main stages (Figure 1). The first stage corresponds to the thermal expansion of

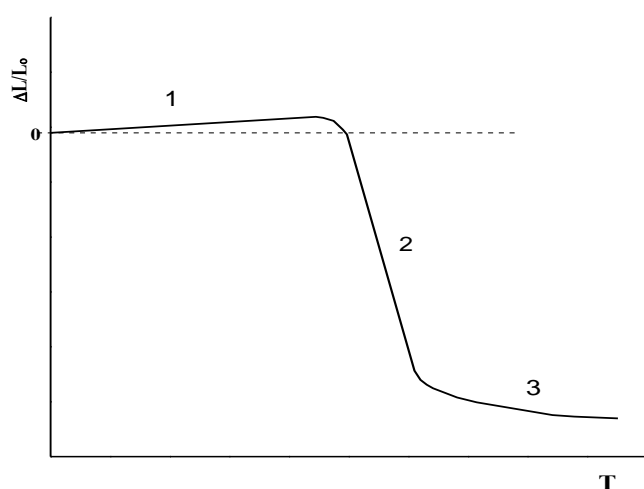


Figure 1 Classical view of the dilatometric sintering curve.

the line with a small positive inclination. Sintering (shrinkage) during this stage does not take place up to nearly the threshold temperature. The second stage starts at the temperatures above the threshold one. This is the most important step, when the main and the fastest sintering (shrinkage) of the powder matrix takes place. In the case of monomodal powders, the second region does not have any peculiarities. In the case of bimodal powders with two maximums of the particles distribution, a step appears in this region.

The third stage is a continuation of the sintering caused by the high temperature atomic diffusion. Sintering during the third stage results in the dense ceramics formation usually requires temperatures of the 2/3 orders of the melting points [4].

At the synthesis of dense ceramics, the greatest attention should be given to the second stage. If there is a fault in the realization of this stage, it will be impossible to obtain dense ceramics only during the third. This is the case, when after sintering porous samples with small shrinkage of several percent are taken from the furnace.

The process of fast sintering during the second stage usually proceeds in the temperature range of 300–400 °C. The temperature of the second stage start may be just a third part of the melting temperature, for example it is 500 °C for SnO_2 [1] ($t_{melting} = 1630$ °C) or about 900–1000 °C for ZrO_2 and CeO_2 based ceramic materials [3] ($t_{melting} \approx 2700$ °C). If the expansion of the temperature interval of the second stage and shift of its beginning towards higher temperatures are observed, these facts denote the presence of obstacles in the process.

In a particular case of sintering of multilayer ceramic devices made of different materials, it is important to consider both the closeness of the coefficients of thermal expansion, values of the finite shrinkage of each material, and location of the regions of fast sintering. The sintering temperatures of these regions should differ significantly. To eliminate the deformation of thin flat items, the item for sintering should be placed between two porous plates with possible fillers on the perimeter; the thickness of the fillers should slightly exceed the thickness of the target product.

It is clear that fast shrinkage of the powder matrix during the second stage at low atomic diffusion may take place if the mass transfer proceeds in blocks. Therefore, blocks (particles) should slide relative to each other. To evaluate the rate of this process, Gueguzin [4] in his model of “diffusion sliding” analyzed the variant, when a flat particle is retracted into the opening between two other particles due to the decrease in surface energy at fast surface diffusion. The calculation was performed at the uninhibited sliding. The particle retraction time was equal

to several seconds at 1000 °C and rational constant values. Such a process rate is 10^5 times faster than at the atomic diffusion of the substance from the particle volume to the region of the passage.

At an ordinary sintering, such a high sliding rate is not realized due to the presence of rough edges on the sliding surfaces. It is obvious that sliding of the rough particles relative to each other is facilitated if the contact areas are minimal, as it is in the case of spherical particles that are used for the formation of dense ceramics at low temperatures. However, in the ensembles of spherical particles, the retraction does not take place.

The peculiarities of the particles sliding in the powder ensemble become understandable when we consider the fact that the powder particles are rather phonon oscillators, rather than static objects. That is why each particle in the powder ensemble vibrates. The vibrations increase proportionally to the temperature and when the temperature threshold is reached, the interparticle bounds are interrupted and sliding takes place. Starting from this moment, a fast shrinkage of the powder matrix, contracted by the forces of the surface tension, is observed (Figure 1).

The sliding powder particles move and pack up to a certain density limit. It seems that the formed porous powder structure should sinter into dense ceramics only during the third stage. In the majority of cases that is what happens. However, dense ceramics is formed during the second stage as well if the powders composed of mono-sized spherical particles are used. For example, in papers by Barringer and Bowen [1, 5, 6], the TiO_2 based ceramics with the density exceeding 99 % were obtained from almost spherical mono-sized powders from two batches (0.1 and 0.4 μm) at the sintering temperatures of 800 and 1050 °C for 90 minutes, respectively.

The classical sintering model does not clearly define the conditions of the particles being mono-sized because in the package of similar balls big empty areas, i.e. pores, will exist. That is why according to the classical model it is advised to add fine fractions to fill these areas. This recommendation works, but only under the condition of high-temperature sintering, when the atomic diffusion takes place.

The ideal hexagonal package of similar balls has a density of 76 %. Hence, the maximum achievable density of the ceramics during the second stage could not exceed this value. However, fast sintering at low temperatures may result, as it is seen, in the formation of almost 100 % dense ceramics from the mono-sized spherical particles. This means that apart from sliding there is a different process that results in the pores filling.

To explain such dense sintering without considering atomic diffusion, Gueguzin [4] suggested that the powder particles during the sliding turn and adjust to each other, or even change the form and fill in the interparticle distances. However, this suggestion is not true for the mono-sized spherical particles of the powder, with which the maximum density is reached during the second stage. Empty spaces between similar spheres will remain in any case, independent of the sphere location. The way the particles change their form is also not clear.

The model of vibrating particles allows answering this question. Indeed, each particle of the chemically obtained powder ensemble is always composed of the smaller items. This is even more true for spherical particles. The spherical shape directly elucidates that the particles are composed of fine nanoparticles. Otherwise, these spheres could not have been formed. We would not have observed spheres but faceted grains.

Further, the vibration of powders pressed against each other with the surface tension forces will move the surface nanoparticles in the contact region. Nanoparticles will start moving towards the empty space. In addition, the maximum vibrations of powders will be observed exactly at the mono-sized spherical particles because the resonance appears due to the phonon vibration spectra. The sliding of powder particles and the nanoparticle movement rate will noticeably increase during the resonance. Therefore, fast resonance ceramic sintering will take place.

If powder particles are not composed of fine nanoparticles, and have a structure of dense grains than the above presented process cannot take place. When the sliding and packing processes are finished, dense ceramics may be obtained only during the third stage by atomic diffusion. That is why dense ceramic materials composed of slicker, prepared by grinding of sintered present samples, are obtained only by the high temperature sintering.

Therefore, resonance sintering of the mono-sized spherical powders is the fastest method of dense ceramics production at the lowest temperatures. However, the synthesis process of such powders and the formation of target products are difficult tasks that do not simplify the dense ceramics formation. This process is promising for the production of nanoceramics with desired properties.

Nevertheless, the phonon vibrations of particles and thermal process always takes place. That is why the stage of fast sintering, caused by vibrating particles sliding, is observed to a greater or lesser extent in the particles of other forms, which may be obtained easier.

Therefore, let us consider the conditions that simplify or vice versa hinder the fast sintering process according to the suggested vibration model of particle sliding.

3. Ultra-high frequency induction sintering

According to the above model, if any powder ensemble (particle form may be different) is additionally to the proper particle vibrations is subjected to the outer high-frequency vibrations, the powders sliding facilitates and the process rate of low temperature sintering increases. Indeed, at the ultra-high frequency induction heating of the powders (usually at the frequency of 2.45 GHz, but sometimes the frequencies up to 85 GHz may be used) the shrinkage rate may reach exceptionally high values, up to 3 % per minute [1]. These rates are comparable with the rates calculated by Gueguzin [4] for the unhindered sliding.

For instance, Rahaman [1] provides the data of Janney and Kimrey on ultra-high frequency induction sintering at 28 GHz of the MgO doped Al₂O₃ powder samples. After the ultra-high frequency induction heating at 1000 °C for 1 h, the sample density reached 95 %, when at the common sintering at the same temperature, the sample density was 57 % and scarcely exceeded the density of the powder sample of 55 %. In addition, the sintering activation energy decreased greatly to 170 kJ/mol, when for the common sintering it was equal to 575 kJ/mol. Such an abrupt difference in two processes denotes that ultra-high frequency induction activates the vibration sliding of a particle, not just the atomic particle vibrations, and their diffusion.

4. Agglomeration

Tendency of the system towards the minimum of the surface energy is the thermodynamic basis of the powders sintering. The activity of powders towards sintering related to the mass unity may be evaluated by the α factor that is equal to the product of particles number, the averaged surface square of one particle and specific surface energy ω :

$$\alpha = 3\omega/rd, \quad (1)$$

where r is the averaged particle radii, d is the density, g/cm³. That is why it is important to obtain fine powders during the synthesis. Such powders have high specific surface energy, which is an initial condition of their dense sintering.

However, even nanopowders with high surface energy may not solve the problem of dense ceramics formation without the second important condition – the powder particles should have a form that favors sliding,

i.e., the form should be close to spherical. To obtain high density ceramics during the low-temperature sintering the powder particles should be preferably mono-sized.

Agglomerated powders, where the sliding is extremely hindered because of the complex form of particles, are the antipodes of spherical powders. Therefore, the second sintering stage for such powders shifts to the region of noticeably higher temperatures. The process of particles agglomeration will proceed and, as a result, the sintering of high density ceramics will be greatly hindered. Even though low pressing density of such samples is commonly accepted as a reason of poor sintering. There is no doubt that pressing also influences the process, but poor pressing is also caused by slow particles sliding during the powders sample formation.

A widely used method for nanopowder formation by combustion is relatively simple; if the sparkle temperature is high, it results in the agglomeration of the powder. Moreover, the powders after synthesis are additionally calcined to remove organic traces.

If nanoparticles in the agglomerates are strongly bound and this connection is not destructed by the ultrasound, then the formation of dense ceramics from such powders is hindered. The grinding does not solve this problem, because during the common grinding in a ball mill the particle size remains larger than 1 μ m. That is why the samples are sintered in the same way as other powders with a particle size nearly equal to that of agglomerates [1]. This idea is verified in a review [5], in which data on the LaGaO₃ based electrolyte sintering at 1400 °C obtained in 19 different sources are compared. Ceramics obtained by a solid-phase synthesis had a density of 98 %. Ceramics prepared by more complex chemical methods using a preliminary calcination of powders at 1200 °C did not show better characteristics.

Let us note that in the solid phase method, a preliminary synthesis in pressing forms with the further grinding allows the formation of dense ceramics. If synthesis is performed in powder, which seems to reach high disperse values easily, the dense ceramic samples are not obtained. The reason is simple. The calcined powder has agglomerated particles and at the grinding of the sintered pressed form, relatively dense spherical particles are formed.

As we see, α factor and the particles form are completely determined by the powder synthesis method. That is why different powders composed of the same materials may have different sintering properties.

5. Influence of admixtures and adsorbed gases

The sliding model suggests that any reason that influences the conditions of the sliding particles surfaces

will influence the process of the fast sintering. Surface is a complex object and in some cases, based on the experience, even negligible amounts of admixtures (not exceeding hundredths of percent) may have a decisive influence on the sintering. The presence of admixtures at the surface may either hinder or accelerate sintering. In the last case, the admixtures are added as sintering agents, it is reported in [8]. Particles sliding during the fast sintering stage may be significantly facilitated by easy melting additions. However, sintering agents are not always appropriate for the synthesis of functional ceramics and ceramics with required satisfactory mechanical characteristics at high temperatures.

Another reason that influences the surface conditions is a gas phase being in contact with the particles surface, both during the storage and calcination. That is why the characteristics of the gas phase and the time of contact with a highly disperse powder will definitely influence the state of the surface and its sintering. This is especially important for the powders that contain water sensitive elements. When working with such materials, the rule is to sinter the ceramics from the powder directly after its synthesis. Even a short-time storage of such powders may have a detrimental effect on the sintering. So, to say, the synthesized powder and the stored for 24 h in a desiccator synthesized powder are absolutely different. This empiric fact has been long stated by analytical chemists. When determining the losses at the heating and cooling the crucible with the powder, the process in the desiccator should take the minimum required time. Any longer exposure causes the increase in the sample weight; it equalizes with the dehumidifier.

The repeated calcination of the powders to activate them and to remove the chemisorbed water may not give significant results. It appears that the initial hydration inevitably changes the topography (roughness) of the powder particles and sliding becomes more difficult.

The form of the first region of the dilatometric curve allows us to conclude that there are processes that hinder the sintering. The region should be straight, and there should not be any peculiarities. The deviation downwards denotes the loss of the hydrated water, or the continuation of the precursor decomposition. The upwards deviations are the most undesirable, even if they are small. This fact denotes the surface loosening due to the interaction between the powder and the atmosphere water vapor. Therefore, the fast stage will be hindered, it will be either shifted to the region of higher temperatures or even blocked. In any case, the sintering of dense ceramics will be problematic.

We are facing this problem when synthesizing proton-conducting oxide materials; their sintering

appeared to be sensitive to the water vapors present in the atmosphere. That is why synthesis was more successful in the winter period rather than in summer time with humid air. The synthesis problem was solved when we considered the factor that is usually missed even by highly experienced ceramic researchers. The fact is that the furnace lining adsorbs water. When heated water desorbs and humid atmosphere is created in the furnace, which is harmful for the samples. The furnaces blowing with dry air via zeolites is not effective, because of the lining size. Then the firing was performed using furnaces with SiC heaters; the temperature of the furnace was maintained at ≈ 700 °C that prevented moisture absorption by the lining. The sintering methodology with the introduction of pressed forms of the recently calcined powders directly to such a hot furnace allowed, for example, obtaining ceramic samples based on BaCeO_3 and SrCeO_3 of the 97–98 % density at the sintering temperature of 1450 °C for 2 h.

Apart from the water vapors, highly disperse powders absorb other atmospheric gases and residual gas components of the chemical synthesis reaction. This stage of fast sintering is not supposed to be accompanied with the gases evolution, as they might form pores. That is why before the fast sintering, the powders should be exposed to remove all gases from the sample. For example, just a 30-minute exposure at 900 °C of the LaGaO_3 based samples (50 °C before the initial stage of the fast sintering) allowed obtaining by the solid-phase synthesis ceramic samples with the stable density of 96–99 % at 1400–1500 °C. Without this short degasification, the high density of ceramics was not achieved.

The continuous heating was found to be more efficient at thin samples sintering; these samples have a faster degasification during the heating. The surface of thick samples is frequently smooth and dense, but if there are pores inside them, this indicates problems with degasification.

To improve the sintering process, significantly longer preliminary exposures, reaching tens of hours, are performed [1]. It is clear that such long-term exposures are related mainly with the processes of surface diffusion rather than powder matrix degasification. From the thermodynamic point of view, such diffusion will smooth the surface of the powder particles, facilitating their sliding.

Probably, in the number of cases these long-term exposures before the fast sintering stage can improve the powders, which surfaces became rough after the interaction with water vapors, and, hence, allow forming dense ceramics from them.

Rahaman [1] has repeatedly noted that achieving high ceramic density becomes more difficult if the grain growth process prevails over the compaction process. However, the question arises, why does diffusion result in the grains enlargement rather than in ceramics packing, if the process of porosity decrease is more beneficial from the energy point of view? The insufficient ceramics degasification might be the reason. Therefore, the formed pores cannot close because of the high gas pressure in them. This is supported by the idea that the predominance of the enlargement process over the packing one is characteristic for the covalent types of carbides and nitrides [1], where gas diffusion is hindered.

6. The influence of the heating rate

The recommendation to burn ceramic products slowly comes from the depths of centuries, when pottery began to be made. It is true for some cases. First, if material is hydrated, as in the case of clay, or contains organic binding, the sample should be heated slowly. Secondly, during the sintering of large items, fast heating will cause a large temperature gradient in the object, which may result in its deformation and cracking.

Nevertheless, based on the experience, fast heating results in the higher density of the ceramics [1]. The model of vibrational sliding explains the need for a high heating rate for dense ceramics formation. As stated above, the stage of fast sintering proceeds in a definite temperature range (Figure 1). The presence of this range denotes that in the powder matrix there is a number of particles of different mobility. The particles mobility in the powder matrix is mainly defined by the number of its contacts with the environment. The greater number of contacts results in lower mobility and high temperature of the particle sliding. That is why two main processes proceed in the matrix. The particles with initially large number of contacts will remain on their places for a long time and will not slide at the temperature growth, which causes sintering of such particles with each other and formation of agglomerates.

The particles with a small number of contacts will be the first to move, when the critical temperature is reached. They will deposit in the places, where the number of their contacts increase; i.e., in the places with large particles accumulation. As a result, at slow heating the powder matrix transforms into the ensemble of dense agglomerates with large irregular pores between them. This is well seen during our experiment on the example of the Y_2O_3 powder sintering with spherical particles (Figure 2). The same picture is observed for the spherical powders of complex compounds, such as $Gd_2Zr_2O_7$ pyrochlore [10].

Hereof it follows that if after degasification the sample is rapidly heated to the temperature corresponding to the end of the fast sintering stage, then, ideally, all particles will start sliding simultaneously and the sample will not agglomerate and will reach maximum density in the short period of time. The short sintering time is favorable for obtaining a fine crystalline structure.

Such extra fast sample heating may be performed, when the sample is placed in the heated to the desired temperature area of the tubular furnace [1]. However, this strict regime may be used only with small-sized samples due to the danger of appearance of surface sintering cracks.

Let us pay attention to one more peculiarity of the fast heating. Evolution of the surface energy at the sintering of highly dispersed powder will increase its temperature. At slow heating, this heat spreads and does not have a significant influence on the sintering process. At rapid heating, the sample temperature may increase noticeably. Moreover, the surface energy from the microscopic approach will be transmitted to the surface atoms (surface nanoparticles) and not to the entire sample. Therefore, the surface structures will abruptly increase their motion. As a result, fast heating provides fast sliding of all particles, which decreases their agglomeration, and further sintering of the particles between each other.

The increase in the sample temperature will be especially significant at the reaction sintering, which is accompanied by the evolution of a significant heat of reaction. To obtain dense ceramics during this process, the diffusion coefficients of the cations should be close enough to eliminate the Kirkendall effect that causes the pore formation.

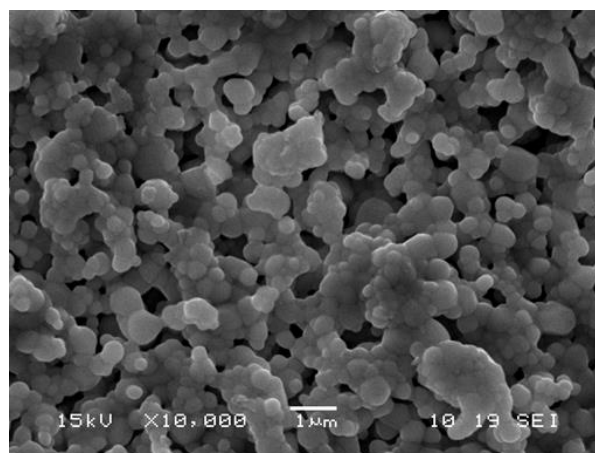


Figure 2 Microphotograph of the powder matrix with the Y_2O_3 spherical particles (the grade for luminophores is 99.992 %) after heating with the rate of 100 °C per hour up to 1450 °C and 8 h exposure.

The effect of the reaction sintering has been observed in our practice in the synthesis of $\text{BaCe}_{0.9}\text{Nd}_{0.1}\text{O}_{3-x}$ ceramics. The pressed form of 14 mm in diameter contained the mixture of initial BaCeO_3 , CeO_2 and $\text{NdO}_{1.5}$, components. It was put into the heated furnace at $900\text{ }^\circ\text{C}$ and exposed for 30 minutes. The obtained dark brown ceramics was dense without any cracks and the fractured part had a glass-like glitter. It was obvious that the sample temperature was significantly higher than $900\text{ }^\circ\text{C}$, whereas the significant CO_2 gas evolution did not cause the pores formation. This effect requires further analysis. At the analogous synthesis of the SrCeO_3 -based ceramics, the sample, in contrast, increased greatly in size and was very porous.

7. Conclusions

The dilatometry curve of the powder matrix sintering elucidates three stages of the sintering process. The stage of fast sintering at low temperatures (second stage), when the rate of the atomic diffusion is small verifies the “particles sliding” model, according to which the shrinkage proceeds via the powder particles sliding. However, the simple sliding model does not explain the main facts: formation of dense ceramics during this stage and required mono-sized spherical particles for dense ceramics formation.

The suggested model of phonon vibration of the powder particle matrix, which from the same point explains the experimental facts such as the resonance sintering of mono-sized particles. In the present work the conditions and factors that influence sintering have been analyzed.

The sintering processes have been observed using the example of oxide ceramics, the common principles of this model may be applied for the sintering of all ceramic materials.

Supplementary materials

No supplementary materials are available.

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Author contributions

Valery Gorelov: Conceptualization; Data curation; Formal Analysis; Writing – Original draft; Investigation; Data curation; Methodology; Project administration; Supervision; Writing – Review & Editing.

Conflict of interest

The authors declare no conflict of interest.

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