Dynamic recrystallization and deformation constitutive analysis of Mg–Zn-Nd-Zr alloys during hot rolling

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ABSTRACT
Due to limited basal slips, the recrystallization of Mg–Zn-Nd-Zr alloys originated at grain boundaries with large orientation gradient based on strain-induced-boundary-migration nucleation mechanism. The primary driving force of the recrystallization was the distortion energy difference around grain boundaries. However, more non-basal slips occurred near the grain boundary at larger strain deformation, so that some dislocations of adjacent subgrain boundaries with less orientation difference transferred to other subgrain boundaries by means of dissociation and disassembly, which is called the subgrain boundary merging. Meanwhile, some ductile shear zones appeared surrounding the primary grain, which held multiple slip systems moving around the grain boundary, gave rise to larger orientation gradients and facilitated recrystallization grains growing.

1. Introduction

The low ductility due to the absence of effective slip systems greatly limits the application of magnesium alloys. Grain refinement, as known as a feasible means to eliminate this defect, has been attracting more and more attention [1, 2, 3, 4]. There is no doubt that dynamic recrystallization and static restoration, which occurs in magnesium alloys during thermal machining and subsequent heat treatment process, will result in great grain refinement. Studies showed that the additional slip system in hcp crystal structure was more likely to be activated at elevated temperature, which promoted the formability improvement of magnesium alloy [5, 6, 7]. However, the thermal deformation of magnesium alloys at high temperature was often accompanied by extremely severe oxidation overfiring and grain coarsening due to its low thermal stability. Therefore, careful determination of the optimum temperature range for magnesium alloy thermal processing was essential [8, 9, 10, 11]. Incorporation of some microelements could effectively reduce the grain size of magnesium alloy and regulate the recrystallization texture to improve the plasticity. In particular, rare earth (RE) elements could not only improve the formability through texture modification, but also significantly improve the high-temperature stability of magnesium alloy.

In this regard, neodymium (Nd) containing magnesium alloys received particular attention due to their excellent strength and ductility [12, 13, 14, 15, 16].

At present, the research on thermal deformation of Nd containing magnesium alloys mainly focused on magnesium alloys with high content of Y, Zr, Ag and Zn, mainly due to the mechanical strengthening of these alloying elements. However, studies on thermal deformation behavior of multi-component Mg–Zn-Nd-Zr alloys have not been widely reported [17, 18, 19, 20, 21, 22, 23, 24, 25, 26]. The common characteristic of these alloys with trace alloying elements is that the microstructure changes significantly after thermal deformation. Due to the inhibition of dynamic recrystallization by Nd atoms, the very coarse grain structure of these casting materials usually becomes partially recrystallized, which contains a large number of ultrafine recrystallized particles and some elongated coarse grains. These microstructures make the alloys suitable for thermal deformation and prone to further deformation without significantly reducing their mechanical strength. In a recent study report, the high-pressure torsion (HPT) deformation characteristics of the Mg–Nd alloys was studied [27]. It was shown that the processing by severe plastic deformation using HPT lowered the recrystallization temperature and lead to a decreasing stored energy with
increasing turns. In another report, the deformation behavior and microstructure evolutions of hot rolled Mg–Nd sheets were investigated through intragranular misorientation analysis and quasi in-situ electron backscatter diffraction measurements, where zinc element showed a pronounced texture weakening with a formation of the TD split component during recrystallization [28]. On the basis of these studies, various models were proposed to characterize the thermal deformation behavior of metal materials [29]. Due to the limited data on thermal deformation of Mg–Nd alloys as mentioned earlier, it is of great significance to investigate thermal deformation and its dynamic recrystallization behavior of multicomponent Mg–Zn-Nd-Zr alloys. We focus on analyzing the effects of strain, strain rate and temperature on the deformation behavior of Mg–Zn-Nd-Zr multicomponent alloy during hot rolling and exploring the mechanism of recrystallization nucleation and its growth. Moreover, the constitutive relation of high temperature plastic deformation is expected to be established by calculating the stress value and activation energy of the multicomponent alloy under high temperature deformation.

2. Experimental procedure

The Mg92.2Zn6Nd1Zr0.8 alloys were prepared by ingot metallurgy. First, the melting process was carried out in an electrical furnace under a covering flux and argon gas shielding atmosphere. Then, the billets were homogenized at 673 K for 6 h and then cut into sheets with 16 mm thickness. Last, the sheets were rolled at the temperature precisely controlled from 593 K to 693 K with variable strain ratio. In order to counteract the temperature drop of the alloy during rolling progress and improve the accuracy of temperature control, the rollers temperature were controlled from 423 K to 523 K. The microstructure of the materials, before and after hot deformation, was investigated using optical microscopy (OM), scanning electron microscopy (SEM), and electron back-scattered diffraction (EBSD). To reveal the microstructure, the samples were mechanically polished with emery paper and abrasive-free cloth, and then etched in an acetic glycol solution. Moreover, the thermal compress simulation was used to determine the true stress-strain curve of the alloy.

3. Results

3.1. Deformation on dynamic recrystallization

Figure 1 shows the microstructures of the alloys hot-rolled at the strain rate of 0.1 s\(^{-1}\) and the temperature of 623K in different strain variables. It is obvious that recrystallization increases with the increase of deformation degree. There were fewer dynamic recrystallization grains in metallography while the strain had not exceeded 0.1, whereas a fewer arched bend distributed unevenly at the grain boundaries in Figure 1(a). While the strain increased to 0.2, a fewer recrystallization fine grains appeared at some grain boundaries or intersections in Figure 1(b). As the strain continued to increase, an abundant of recrystallization grains occurred near and around the grain boundaries. Those recrystallization grains constantly occupied almost the entire region in metallography. Conversely, the initial grains became less and smaller. Meanwhile, the initial grains were elongated when the alloys deformed greatly, and changed from irregular clumps to rhombohedrons, shown in Figure 1(c) and Figure 1(d).

3.2. Temperature on dynamic recrystallization

On the other hand, the growth of dynamic recrystallized grains is closely related to the deformation temperature. The microstructure of the alloy, which was deformed to 0.6 at different temperatures at a constant deformation rate of 0.1 s\(^{-1}\), is shown in Figure 2. When the deformation temperature was below 573 K, the grains of dynamic recrystallization were fine and uniform, shown in Figure 2(a) and Figure 2(b). However, as the deformation temperature rose to 623 K, the dynamic recrystallized grains became a mixture of grains with uneven size, shown in Figure 2(c). Once the deformation temperature exceeded 623 K, the recrystallized grains would rapidly merge and grow into coarse grains, shown in Figure 2(d) and Figure 2(e).

Figure 1. Microstructures evolution with strain ε of (a) 0.1, (b) 0.2, (c) 0.6 and (d) 0.8.
3. True stress-strain curve

The true stress-strain curve of the Mg92.2Zn6Nd1Zr0.8 alloy, shown in Figure 3, were measured by thermal compress simulation, showing that similar dynamic recrystallization behaviors occurred when the alloys deformed between the temperature of 473 K and 723 K under the strain ratio of 0.01s\(^{-1}\) to 1s\(^{-1}\). The change of stress value had four obvious characteristics with the increase of strain. At the first stage, the decreasing flow stress and the strain value corresponding to the first stage, the decreasing flow stress and the strain value corresponding to the second stage, the work hardening effect due to dynamic recrystallization was higher than the work hardening effect. Much more recrystallized grains appeared at grain boundaries and further squeezed the primary grain. In the final fourth stage, the stress softening and the work hardening effect tend to a new equilibrium. The primary driving force of the recrystallization in the critical deformation region is the energy difference of distortion between both sides of grain boundaries. At the beginning of the deformation, the deformation within the alloy is uneven. Due to the uneven deformation, the dislocation density on both sides of the original large Angle interface is different, which results in the difference of local storage energy. The accumulation of Nd and Zn near the grain boundary increased the degree of distortion and promoted more nucleation cores. The subgrains in the low dislocation density region grew into the neighboring ones with high dislocation density to form tongue-shaped swelling subgrains. This bulging subcrystal, which has little strain hardening, then becomes a recrystallized nucleate core. This is called the strain-induced boundary migration (SIBM) nucleation mechanism. The kinetics of recrystallization nucleation conforms to Bailey-Hirsch moulds.

Under the action of Gibbs free energy \(G\), a portion of a grain boundary with diameter \(2L\) is arched with curvature radius \(R\). While the protruding part pushes forward and increases volume \(dV\), and the corresponding interface area increases \(dA\), the Gibbs free energy change \(\Delta G\) can be described as Eq. (1):

$$\Delta G = \left(\gamma_b \frac{dA}{dV} - E\right) dV$$

(1)

where \(E\) is the storage energy difference per unit volume on both sides of arch partial grain boundary, and \(\gamma_b\) is the grain boundary energy.

In order to ensure the arch out process can be realized, \(\Delta G\) should be less than zero. Thus, Eq. (1) can be simplified to Eq. (2):

$$E > \gamma_b \frac{dA}{dV}$$

(2)

Namely, the SIBM nucleation occurs only when the energy difference \(E\), caused by deformation, is greater than a certain value, which is determined by the boundary energy of the material and the shape of recrystallized crystal nucleus.

Assume that the grain is spherical, then we can derive Eq. (3) from geometric relations in Figure 4.

$$\frac{dA}{dV} = \frac{2}{R}$$

(3)

The minimum curvature radius \(R_{min}\) of grain arch should be greater than or equal to \(L\) so that the grain boundary arch moves forward.
spontaneously and continuously. The thermal activation arch size $L$, caused by plastic deformation of the alloy, should be larger than a certain value to become the nucleation core of recrystallization (Eq. (4)).

$$L \geq \frac{2T_b}{E}$$  \hspace{1cm} (4)

However, the origin of the arched low disjunction density region is still unknown. One possibility is that the storage energy difference is too large due to the grain orientation difference, which is related to the rate of dislocation accumulation during deformation, on both sides of grain boundary. Different dislocation accumulation rates lead to the energy storage difference between the two sides of grain boundary, which causes the arch core. Correspondingly, another possible reason is that at one side of the grain boundary, low-density recrystallization subcrystals are preferentially restored to form. These preferential subcrystals arch to the other side with high dislocation density to form recrystallization core.

When the deformation degree of the alloy increased to about 0.6, the recrystallization core couldn’t be nucleated by grain boundary bulking anymore, because the distortion energy is similar on both sides of grain boundary. However, some dislocations of adjacent subgrain boundaries with less orientation difference are transferred to other subgrain boundaries by means of dissociation and disassembly, thus resulting in subgrain boundary merging shown in Figure 6. On this point, much more dislocation slipping and climbing due to larger deformation provide higher dislocation energy for subgrain boundary merging. In fact, all

Figure 3. True stress-strain behaviors with various strain rate of (a) 0.01 s$^{-1}$, (b) 0.1 s$^{-1}$, (c) 0.5 s$^{-1}$ and (d) 1s$^{-1}$.

Figure 4. SIBM recrystallization nucleation: (a) SEM image and (b) the Bailey-Hirsch kinetics model.
kinds of subcrystals began to grow in the crystal recovery stage. However, recrystallization nucleation occurs only when subcrystal growth is accompanied by large angle grain boundary migration. In particular, when the subcrystal polymerizes and coarsens to a certain size, the orientation difference between the subgrain and the surrounding grains reaches about 12° or more, the subgrain boundary becomes the large angle grain boundary [30]. At the same time, it migrates at a greater rate, thus forming a recrystallization core. In other words, orientation gradient is a indispensable condition for coarsening nucleation in subcrystalline polymerization [31].

By contrast, some ductile shear zones appeared at the boundary of the primary grain, in where there were some long strip subgrains nucleating and growing rapidly, shown in Figure 7. The possible main reason is that in large strain deformation, multiple slip systems tend to move around the grain boundary. These slips give rise to larger orientation gradients, thus facilitating recrystallization grains growing.

In conclusion, the existence of large orientation gradient is crucial to recrystallization nucleation no matter the strain-induced boundary migration or subgrain boundary merging. In addition, all new recrystallized grains and its orientation are derived from the subcrystal in the deformed matrix as well as its crystal orientation.

4.2. Variation of recrystallized grains

To further investigate the evolution of recrystallized grain, EBSD analysis was performed on the typical samples with strain of 0.2, 0.6 and 0.8, shown in Figure 8. There is no doubt that the nucleation and growth of recrystallization are greatly influenced by strain variables. According to the SIBM nucleation mechanism and Bailey-Hirsch moulds, the larger strain causes more stress concentration and Gibbs free energy at the grain boundary front, which provides more nucleation opportunities for recrystallization. This deduction can be confirmed by the large number of fine recrystallized grains surrounding the initial grain shown in Figure 8(b) and Figure 1(c), as compared to the number of sporadic recrystallized grains in Figure 8(a) and Figure 1(b). Furthermore, the variation of maximum texture strength, by comparing with the pole Figure s of Figure 9(a) and Figure 9(b), also supports this correspondence between the amount of recrystallization and the degree of deformation. When the strain reached to 0.2, the initial grain exhibits a typical rolling texture with the maximum strength about 45.94. However, when the strain exceeded 0.6, the maximum texture strength decreased from 45.94 to 13.18, indicating that the occurrence of a large amount of recrystallization weakens the texture strength.

However, the increasing strain variables did not lead to a large increase in the number of recrystallization, nor did it lead to significant

![Figure 5. EDS element distribution map around grain boundary (a) SEM, (b) Mg, (c) Nd and (d) Zn.](image1)

![Figure 6. Merging of adjacent subcrystals](image2)
texture weakening, shown in Figure 8(c) and Figure 9(c). On the contrary, when the strain variable increased from 0.6 to 0.8, not only did the initial grain size no longer decrease significantly, but the recrystallized grain size tended to increase, which meant that dynamic recovery was starting to take over.

To sum up, the hot rolling process of magnesium alloy is an interactive process of dynamic recrystallization and dynamic recovery. At the beginning of hot rolling deformation, the increase of deformation amount leads to stress concentration in grain boundary front. The Gibbs free energy generated by these stress concentrations promotes the nucleation of recrystallized grains. Therefore, a large number of recrystallized grains appear around the initial grain. These recrystallized grains are beneficial to weaken the rolling texture and reduce the adverse effect of stress concentration caused by large deformation. At the same time, with the increase of strain variables, the recrystallization nucleation caused by stress concentration becomes slow. In contrast, dynamic recovery at high temperatures gradually increased and became dominant. Once the effect of dynamic recovery exceeds the occurrence of recrystallization nucleation, the average grain size of the alloy will gradually increase.

4.3. Ductile shear of recrystallized grains

As mentioned above, a large number of initial grains gradually changed from irregular lumps to flat rhombic with the increase of deformation degree. To further elucidate the microstructure evolution and dynamic recrystallization mechanism of alloys during hot rolling process, a ductile shear zone model was proposed, shown in Figure 6 and Figure 8(b). In the initial stage of small deformation, the work hardening is greater than the softening caused by dynamic recovery. And with the progress of deformation, the work hardening rate gradually decreases until it is balanced with the dynamic softening. Due to the absence of dynamic recrystallization in the dynamic recovery process, the deformed grains retain their original equiaxial grain morphology. When the strain reaches about 0.2, dynamic recrystallization begins and results in a decrease in the work hardening rate. These dynamic recrystallization
grains are mainly located at the grain boundary of the original grain, shown in Figure 1(b) and Figure 8(a). When the alloy continues to deform to about 0.6, more recrystallized grains appear at the grain boundary of the original grains. Meanwhile, the initial grain is slightly elongated, shown in Figure 1(c) and Figure 8(b). In the process of deformation of polycrystalline materials, the compatibility between adjacent grains leads to the fact that the deformation of each grain must be coordinated by its neighboring grains [32]. This compatibility condition leads to the initiation of multiple slip systems near the grain boundary when the polycrystalline sample deforms. Thus, when magnesium alloy is deformed at high temperature, these grain boundaries slip and lead to microscopic plastic flow near the grain boundaries. Meanwhile, due to the limited number of basal slips and the only {1012} twins, cross slip and climbing are not easy to occur in the deformation process of magnesium alloy. As a result, the magnesium alloys form the typical basal texture with {0001} basal plane perpendicular to the rolling direction TD during rolling process [30]. Simultaneously, some shear bands appear rapidly across the alloy, shown in Figure 8(b).

These massive recrystallized grains near the grain boundary move and change their relative positions through sliding and diffusion coordination. A sinusoidal curve simulation interface reveals the diffusion coordinated steady state grain boundary sliding process, and draws the conclusion that the sliding and diffusion flow act together when a single grain boundary moves under applied stress [33]. Ashby-Verral model confirms this conclusion and argues that the grain slip coupling changes in fine shape under the influence of stress. And this change follows a certain sequence and is accomplished by relatively small strains within the grains [24].

Although these shear zones are prone to strong local shear deformation, which may also be the source of crack, the interaction between dynamic recovery and recrystallization during hot rolling processing makes the zones some easily ductile zones. Of course, these ductile zones are also likely to be beneficial to improve the plasticity of the alloy during the hot rolling process. Therefore, the shear bands or other deformation inhomogeneous regions in the deformation of the magnesium alloys play an important role in the control of the deformation structure.

4.4. Constitutive analysis

4.4.1. Derivation of constitutive equation

To further understand the plastic deformation characteristics of alloys, the relationship between the peak stress $\sigma$ of high temperature plastic deformation and strain rate $\dot{\varepsilon}$ and deformation temperature $T$ was tested, shown in Figure 10.

It is obvious that all curves show a regular linear relationship. Therefore, we use an equation to fit the function of peak stress to explore its constitutive equation with strain rate and deformation temperature. Previous studies have shown that the hyperbolic Arrhenius model is suitable for analyzing the constitutive relationship of magnesium alloys in a wide temperature range. Therefore, the constitutive model of strain rate $\dot{\varepsilon}$, steady flow stress $\sigma$ and deformation temperature $T$ was established based on Eq. (5) [35, 36].

$$\dot{\varepsilon} = A[\sinh(\alpha \sigma)]^n \exp \left(\frac{-Q}{RT}\right)$$

(5)

where $Q$ is the deformation activation energy, which depends on the deformation mechanism of the alloy at different temperatures. R, A, $\alpha$ and $n$ are universal material constants.

Then the formula of function relation between constant $n$ and deformation activation energy is deduced, as follow Eqs. (6) and (7):

$$n = \frac{\partial \ln \dot{\varepsilon}}{\partial \ln [\sinh(\alpha \sigma)]} \bigg|_T$$

(6)
The deformation activation energy was calculated using the plots in Figure 10 and the relationship between deformation activation energy and temperature was drawn at different strain rates in Figure 11.

All the curves show the same trend: The deformation activation energy increases gradually with the increase of temperature from 573K to 673K, correspondingly, increases rapidly with the increase of temperature from 673K to 723K. This indicates that the thermal deformation mechanism of the Mg92.2Zn6Nd1Zr0.8 alloy is variable according to different temperature and strain rate range. Further research shows that when magnesium alloys deform at lower temperature, basal slip and twin coordination are the main drivers of continuous deformation of the alloys because of the higher CRSS value of non-base slips [37]. However, the decrease in CRSS values of non-base slips leads to an increase in the number of coordinated dislocations slips at higher temperatures. Moreover, when the c-axis orientation of the initial grain changes from the...
parallel to the vertical orientation to the compression direction as the transformation progresses, it is also advantageous to start the non-base slips [38]. As a result, the dislocation slip becomes the main deformation mechanism of magnesium alloys.

The relationship between flow stress, strain rate and temperature were quantified by Zener-Hollomon parameter method, and the constitutive relation Arrhenius equation of flow stress, strain rate and temperature were constructed (Eq. (8)).

$$\sigma = 56.95 \ln \left( \frac{Z}{4.66 \times 10^{12}} \right)^{\frac{1}{5}} + \left( \frac{Z}{4.66 \times 10^{12}} \right)^{\frac{1}{3}} + 1$$

(8)

Where $Z$ is Zener-Hollomon parameter.

$Z = i \exp \left( \frac{Q}{RT} \right)$

4.4.2. Deviation analysis

The deviations between the predicted value of the constitutive equations and experimental measurement data of Mg92.2Zn6Nd1Zr0.8 alloys are compared and analyzed to verify the applicability of the constitutive model under the corresponding deformation temperature and deformation rate based on a method of mean relative error $E$ (Eq. (10)) and model correlation coefficient $R$ analysis (Eq. (9)):

$$R = \frac{\sum_{i=1}^{N} (C_i - C) (T_i - T)}{\sqrt{\sum_{i=1}^{N} (C_i - C)^2} \sqrt{\sum_{i=1}^{N} (T_i - T)^2}}$$

(9)

$$E = \frac{\sum_{i=1}^{N} |C_i - T_i|}{C} \times 100\%$$

(10)

Where $C$ is experimental measurement data, $T$ is the predicted value, and $N$ is the number of data.

Through calculation and comparison, we find that the maximum relative deviation between the predicted value and the measured data is 9.89%. Meanwhile, all the data points fall within the 10% deviation and negative deviations lines, shown in Figure 12. On the whole, the predicted value is in good agreement with the measured data as measured $R$ squared is about 0.9733 and $E$ about 4.76%.

4.4.3. Nd reducing deformation activation energy

Studies confirmed that the deformation mechanism of magnesium alloy during hot compression is closely related to the deformation temperature [39]. When the hot compression temperature of AZ31 magnesium alloy was lower than 473 K, deformation twins tended to occur and become the dominant deformation mechanism. However, when the hot compression temperature was higher than 573 K, the deformed microstructure presented typical dynamic recrystallization characteristics. Accordingly, the activation energy of AZ31 was calculated to be 94.4 kJ/mol at low temperature and 127.5 kJ/mol at high temperature based on the above Arrhenius formula calculation. Similarly, ZK61 and ZM61 alloy also exhibited a similar activation energy mutation and two-stage deformation mechanism during hot compression [40, 41]. It is not difficult to infer that the mutation of activation energy at 673 K in Figure 11 should be the theoretical characterization that the deformation of the alloy shifted from deformation twins to dynamic recrystallization dominated by dislocation slip, cross slip and climb. However, at the base deformation rate of 1.0 s⁻¹, the average activation energy of Mg92.2Zn6Nd1Zr0.8 alloy at low temperature was about 128.04 kJ/mol, which was much lower than 149.83 kJ/mol of Mg94Zn5.2Zr0.8 (ZK61) alloy without Nd element. This means that the deformation twins are more easily activated and generated by adding Nd element. Moreover, at the same strain rate, the activation energy of Mg92.2Zn6Nd1Zr0.8 alloy at 723 K was 168.94 kJ/mol, which was also significantly lower than that of ZK61 alloy with about 198.68 kJ/mol. It is obvious that the addition of Nd element plays a significant role in reducing the deformation activation energy of magnesium alloys during hot working, which is beneficial not only to the twin deformation at low temperature, but also to the dislocation slip, cross slip and climb at high temperature, thus improving the plastic deformation properties of magnesium alloy.

5. Conclusions

(1) The recrystallized grain size and volume fraction of the magnesium alloy increase with the increase of deformation temperature as the strain rate is constant. Meanwhile, the recrystallization recovery and softening effect are enhanced, and the flow stress of the alloy decreases. However, the deformation resistance increases with the increase of the strain rate, which is attributed to the fact that the dislocation does not have enough time to cancel each other through slip, cross slip and climb.

(2) Recrystallization often nucleates in the original grain boundary at large strain. Without a directional gradient, recrystallization does not occur even with large deformations.

(3) The ductile shear zones hold multiple slip systems moving around the grain boundary. As an inhomogeneous deformation region, it is not only beneficial to improve the plasticity, but also the origin of cracks, so the microstructure control in this area is an extremely important means to improve the properties of the magnesium alloys.

(5) The deformation mechanism of the alloy varies with the temperature of hot rolling. At lower temperature, the coordination between mechanical twinning and dislocation slip is undoubtedly the main mechanism to ensure the plastic deformation of the alloy without fracture. With the increase of deformation temperature, more non-base slips provide the conditions for harmonizing the plastic deformation of the alloy.

Declarations

Author contribution statement

Fanxiao Kong: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Yanmei Yang: Performed the experiments; Wrote the paper.

Hao Chen: Conceived and designed the experiments; Analyzed and interpreted the data.

Haiyan Liu, Chao Fan: Contributed reagents, materials, analysis tools or data.

Weidong Xie: Conceived and designed the experiments; Performed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Guobing Wei: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

Data will be made available on request.
Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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