SCIENCE CHINA
Physics, Mechanics & Astronomy

• Article •
Special Issue: Physical Mechanics

June 2012 Vol.55 No.6: 1051–1058
doi: 10.1007/s11433-012-4743-4

The influence of agglomerates on the densification and microstructural evolution in sintering of a multi-particle system

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Received December 13, 2011; accepted February 17, 2012; published online April 27, 2012

Effects of agglomerates on the densification behavior and microstructural evolution during solid-state sintering of a cube of copper particles have been studied with discrete element method (DEM). It is found that the densification of the sintering system decreases as the volume fraction of agglomerates increases. At a given volume fraction of agglomerates, the smaller the size of agglomerates, the poorer the densification and more inhomogeneous the compact is. The morphology and distribution of agglomerates have negligible effects on the densification, especially for the case with a low volume fraction of agglomerates. Agglomerates with a smaller average coordination number would have more restriction on the densification of sintering bodies. To our best knowledge, it is the first time to study the effect of agglomerates on sintering behavior using DEM. This study should be useful for further investigations of the effect of various inhomogeneities of microstructure on the complex sintering process by DEM.

solid-state sintering, discrete element method, agglomerate, microstructure evolution

PACS number(s): 81.05.Rm, 81.40.Gh, 02.70.Ns

Citation: Wang C, Chen S H. The influence of agglomerates on the densification and microstructural evolution in sintering of a multi-particle system. Sci China-Phys Mech Astron, 2012, 55: 1051–1058, doi: 10.1007/s11433-012-4743-4

Productions of nearly pore-free ceramic and alloy materials with uniform microstructure and high strength are essential in high-performance applications. However, it is often difficult to achieve such productions due to many microstructural inhomogeneities [1] existing in compacts, such as pores, regions of non-uniform particle packing and agglomerates. Agglomerates are a kind of major inhomogeneities in compacts [2], which are regarded as particle clusters in local volumes with larger densities than their surroundings. Agglomerates often form due to inter-particle attraction forces like Van der Waals forces, especially for fine particles [2,3], or calcination [4], capillary forces in wet particle processing [1]. They are also produced purposely for a better flowability to uniformly fill a die cavity in dry pressing [1].

Different agglomerates have different behaviors in compaction. Some "soft" agglomerates collapse into smaller fragments or particles by high pressure compaction, e.g., die pressing or cold-isostatical pressing, while other "hard" agglomerates are strong enough to remain and persist even after sintering. These kinds of strong agglomerates are well-recognized to be detrimental to compaction and sintering even with a very small fraction of them in compacts. They would induce inhomogeneous microstructures with non-uniform pore size distributions and coarse pores in the sintered body [5]. Crack is often developed during sintering due to the different volume shrinkage rates of agglomerates with different initial packing densities [1,6–8]. Additionally, grain growth is more significant in agglomerated volumes since the grain growth rate is directly related to the density [9,10]. A series of experimental research studies have been dedicated to studying the effect of agglomerates, including

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the volume fraction, strength, density, size, etc., on compaction [2,11–13] and sintering [2–4,13–20]. For example, Rhodes [17] studied the effect of agglomerates on sintering of yttria-stabilized zirconia and observed that agglomerates retard almost entire stages of sintering and limit the potential achievement of fine powder toward dense and fine-grained microstructure. Dynys et al. [14] studied the effect of agglomerates on sintering of alumina and found a significant decrease in shrinkage rate under the presence of agglomerates at the early stage of sintering and decreasing sintering rates with increasing agglomerate contents. Tuan et al. [19] also studied the sintering behavior of alumina powder compacts containing alumina agglomerates and found the densification rate is significantly retarded by hard agglomerates. All the experimental research studies have shed light on the effects of agglomerates. However, some limitations still exist. One is ascribed to the high-pressure consolidation techniques widely adopted in experimental studies. When the powder bodies containing agglomerates are consolidated by high pressure compaction, the agglomerates may undergo deformation and collapse into smaller fragments and particles. As a result, it is difficult to determine the real content and size of agglomerates in experiments. In addition, it is impossible to accurately determine the degree of agglomeration in experiments due to the super-complex microstructure, although various definitions have been proposed [14,21] to characterize the agglomeration.

These aspects could be compensated to some degree by numerical simulations. Numerical calculation has become an indispensible tool to study behaviors of materials with inhomogeneous microstructures in complex circumstances, which allows accurate designs of the complex microstructure of materials, such as the size, fraction, morphology and distribution of pores, agglomerates or other phases. Different load forms, such as isostatic compaction, closed die and uniaxial compaction could also be simulated numerically. More importantly, any details of the microstructure of the system could be extracted without any disturbance on simulation processes at any moment of simulations. Investigation of the microstructural evolution of materials under extreme conditions becomes feasible, such as the high temperature.

Discrete element method is a general simulation scheme originally introduced for the study of rock mechanics [22]. In DEM, each particle is modeled as a sphere that interacts with its neighbors through appropriate force laws and its motion is determined from the interactions with all its neighboring particles. As a result, this method naturally takes the granular nature of materials into account and allows the investigation of phenomena at the particle length scale, such as particle rearrangement [23,24]. Many works have proved that the discrete element method is also an effective tool to investigate the behaviors of sintering bodies [23,25–35], such as densification rates [23,24,36], viscosities [23,37], anisotropic sintering [38], constrained sintering [39] and crack evolution during sintering [23,40]. Furthermore, DEM can also be very useful to study the effect of agglomerates on compaction. Martin et al. [11] studied the morphology and strength of agglomerates by DEM and found that the morphology of agglomerates has a clear effect on green density but the strength is the predominant factor affecting green density. Kim et al. [41] investigated the rearrangement of agglomerates and found that systems with rearranged agglomerates have higher packing density than those without agglomerate rearrangement.

In the present paper, DEM is used to study effects of agglomerates on sintering behavior of a cubic compact containing both copper agglomerates and discrete copper particles. The remainder of this paper is organized as follows. In sect. 2, DEM method and the special force law between neighboring particles in sintering are briefly described. A new parameter "variance of distances between neighboring particles" is introduced to characterize the microstructural uniformity and study the effect of agglomerates on microstructural evolution during solid-state sintering. The method for preparing agglomerates with various properties and the generation of compacts containing agglomerates are explained in sect. 3. Sec. 4 will mainly discuss the effect of agglomerates on sintering shrinkage and microstructural evolution, considering different properties of agglomerates, including the size, shape, volume fraction, average coordination number and distribution in compacts. Concluding remarks are given in sect. 5.

1 Numerical simulation method and definitions of parameters

1.1 Interaction force law in sintering

In numerical simulations of the solid-state sintering of a cubic particle body, particles are treated as discrete elements (spheres), which interact with each other at the contacts by a special force law. At each time step, the resultant force of each particle is calculated in order to obtain its acceleration, and then a new velocity and position can be calculated and updated at the next time step.

Similar to refs. [23,24,36,39,40], the normal force N_s and the tangential force T_s acting at the contact of two contacting particles are

$$N_s = \frac{\pi a_s^4}{8\Delta_b}\frac{\mathrm{d}h}{\mathrm{d}t} - \frac{9}{8}\pi R_p \gamma_s, \tag{1a}$$

$$T_s = -\eta\frac{\pi a_s^2 R_p^2}{8\Delta_b}\frac{\mathrm{d}u}{\mathrm{d}t}, \tag{1b}$$

$$\Delta_b = \frac{\Omega}{kT}\delta_b D_b, \tag{1c}$$

where $D_b = D_{0b}\exp(-Q_b / RT)$ is the diffusion coefficient for vacancy transport in the grain boundary with thickness δ_b and activation energy Q_b, Ω is the atomic volume, k the Boltzmann constant, T the temperature, h the indentation depth between two spherical particles, γ_s the surface energy, R_p the radius of the primary particle, a_s the sintering contact radius which grows according to the Coble's model $a_s = \sqrt{2hR_p}$, du/dt the tangential component of relative velocity at the contact and η is the viscous parameter.

The contact force between two spherical particles was obtained by Bouvard and McMeeking [42]. The mass transport mechanisms assumed in eq. (1) consist of the grain boundary diffusion and the surface diffusion. The first term on the right hand side in eq. (1a) denotes the normal viscous force resisting the relative motion normal to the contact interface between two adjacent particles. The second term in eq. (1a) is the sintering force trying to pull two adjacent particles together. The tangential contact force T_s in eq. (1b) opposes the tangential component of relative velocity at the contact.

1.2 Variance of distances

The variance of distances of two neighboring particles, abbreviated as "variance" in the following part of this paper, is used to characterize the uniformity of the distribution of particles in a sintering body. The "variance" of a system at some sintering time is calculated as:

$$\text{Variance} = \sum_{i=1}^{N}(X_i - \bar{X}) \Big/ (N-1), \tag{2}$$

where X_i is the distance of two neighboring particles, $\bar{X}$ is the mean value of the distances of all neighboring particles, N is the total number of pairs of neighboring particles at some moment during sintering.

Each particle in a granular body has several neighboring particles. The distance of a particle and one of its neighbors changes constantly under the resultant force induced by the contacting particles. For a particle, some of its neighbors depart away from it, while some other neighbors come closely, the distances of the particle and all the neighbors will become non-uniform, which results in an increasing local "variance". For a multi-particle system, "variance", as an average parameter, is used to describe the uniformity of the whole system.

The rationality of this parameter to characterize the microstructural uniformity has been proved by the numerical simulation of sintering of a planar layer of copper particles [43], which was used to study factors influencing agglomeration in a three-dimensional solid-state sintering model for copper particles.

2 Simulation model with agglomerates

2.1 Agglomerate preparation

Various types of agglomerates have been prepared in order to investigate the effect of the morphology, size, volume fraction and distribution in compacts, etc., on solid-state sintering. Figure 1 gives a schematic sketch of five agglomerates with different sizes and morphologies.

In order to prepare agglomerates, we first conduct a sintering simulation of an agglomerate-free cubic compact, in which there are 1200 uniform copper particles of radius 50 μm. The length of a side of the cube is 20.2 times particle radius which indicates an initial volume fraction 0.61. The method proposed by Henrich et al. [23] has been used to generate a relatively uniform initial packing. The interaction forces between contacting particles follow eq. (1). The sintering temperature is 1323 K and the tangential viscosity is set to be 0.001. The other physical parameters are listed in Table 1. For such a system with large particles, the microstructure is relatively uniform and no obvious agglomerates form after sintering. However, the density of the system should become larger and larger during sintering due to the sintering forces. From such a sintered system, a locally uniform region with determined radius can be cut out, which is regarded as spherical agglomerate with a determined characteristics, such as the density, the contained number of particles, the radius. Using such a method, different agglomerates can be made, which will be filled into the original simulation model of a multi-particle system later. Though the agglomerates are taken from the system with particles of radius 50 μm, the radius of particles consisting of such agglomerates can be re-defined numerically in a new sintering system, into which agglomerates will be filled to establish an original simulation model containing agglomerates. For example, to produce a new simulation model of a multi-particle system consisting of particles and agglomerates, if the radius of the discrete particle is 40 μm, the one in agglomerate can also be defined as 40 μm.

Table 2 gives the details of agglomerates used in this paper, including the size, morphology, relative density and the number of particles in each agglomerate, which is denoted by N_{in}.

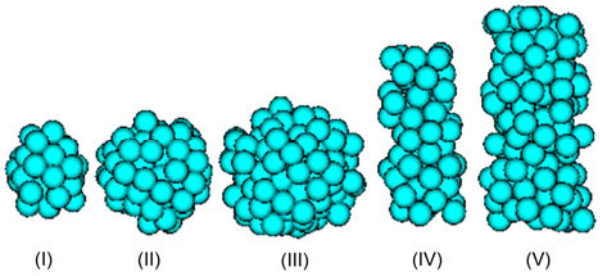

Figure 1 (Color online) Schematic diagram of five agglomerates with different sizes and shapes.

Table 1 Parameters used in the numerical simulation for copper sintering (adopted from ref. [36])

$\delta_b D_{0b}$ (m^3/s)	Q_b (kJ/mol)	γ_s (J/m^2)	Ω (m^3)
5.12×10^{-15}	105	1.72	1.18×10^{-29}

Table 2 Detailed information of agglomerates used in the present paper[a)]

No.	R_{agg}/R_{par}	Morphology	ρ_{rel}	N_{in}
1	3.5	spherality	0.76	34
2	4.29	spherality	0.76	68
3	5.54	spherality	0.76	136
4	6.0	spherality	0.64	136
5	5.0	spherality	0.76	99
6	4.5	spherality	0.76	79
7	4.9/19.0	columina	0.64	136
8	4.8/11.4	columina	0.76	99
9	3.0/14.7	columina	0.76	79
10	2.8	spherality	0.76	20
11	3.8	spherality	0.76	40
12	5.1	spherality	0.76	100

a) For column agglomerates, i.e., Nos. 7, 8 and 9, the two numbers in the second column refer to the non-dimensional radius of the cross section and the non-dimensional height of the column agglomerate using the radius of a particle. N_{in} denotes the number of particles in each agglomerate.

In order to establish a simulation model with agglomerates, a determined number of prepared agglomerates will be put into the region of the initial packing without any resistance, which is ensured to distribute randomly without overlap between any two of them. However, each agglomerate will cover a few existing discrete particles fully or partially. Those particles that overlap too much with an agglomerate, will be removed from the corresponding region. A defined distance D_c is used to justify which particle should be removed. If the distance between a particle P_i in the initial packing and a particle P_j in one of the agglomerates is smaller than D_c, the particle P_i will be removed from the region. One can note that a large D_c adopted will result in a reduced number of particles contacting with the boundary of agglomerates. Therefore, the value of D_c can be used to tune the coordination number of each agglomerate. If not stated specially, D_c is taken as 1.9 to generate the initial compacts with agglomerates in the present paper. Fortunately, we have found that the value of D_c does not influence the final conclusion after a number of testing calculations.

In order to verify the efficiency of the above packing method, Figure 2 gives the initial relative density of the sintering model as a function of the volume fraction of agglomerates, in which the compact contains different number of No.1 agglomerates in Table 2. One can see that the initial relative density decreases linearly with an increasing volume fraction of agglomerates, a similar effect found in experiments for alumina particles [2] and zirconia powders [13]. The linear relation has also been verified using the other types of agglomerates listed in Table 2.

2.2 Simulation model

The simulation model used in the present paper consists of plenty of uniform copper particles of radius 40 μm and agglomerates with a determined volume fraction, which are formed by copper particles of radius 40 μm. The relative density of agglomerate is defined to be 0.64 and 0.76, which ensures the relative density of agglomerate not larger than 0.87 at the final simulation time step. As we know that grain growth is significant at the final stage of an actual sintering and the relative density at this stage is larger than 0.87. However, it is not reasonable any longer to model the sintering body as a packing of spherical particles above this relative density [39,44]. Thus, the relative density of sintering body in the present numerical simulations can not exceed 0.87 and the effect of grain growth is not included. The tangential viscosity in eq. (1) is set to be 0.001, more explanations about which can be found in [23,39,40]. The sintering temperature is set to be 1323 K and all the boundaries of the sintering model are free. The other physical parameters used in the simulations are given in Table 1.

3 Simulation results and discussions

3.1 Volume fraction effect of agglomerates

Figure 3 shows the effect of volume fraction of agglomerates on sintering densification. The volume fractions of agglomerates are 0, 1.7%, 12.1% and 22.5%, which corresponds to zero, one, seven and thirteen numbers of No. 1 agglomerate, respectively. One can see that at the same simulation step, the densification of the sintering body decreases when the volume fraction of agglomerates increases. Similar results can also be found by Dynys et al. experimentally [14], in which the densification behavior of the sintering system consisting of alumina particles of radius 0.5 μm and tens of micrometers agglomerates were investigated and they found the shrinkage rates decrease significantly with an increasing content of agglomerates.

The effect of volume fraction of agglomerates on the microstructural uniformity during evolution is exhibited in

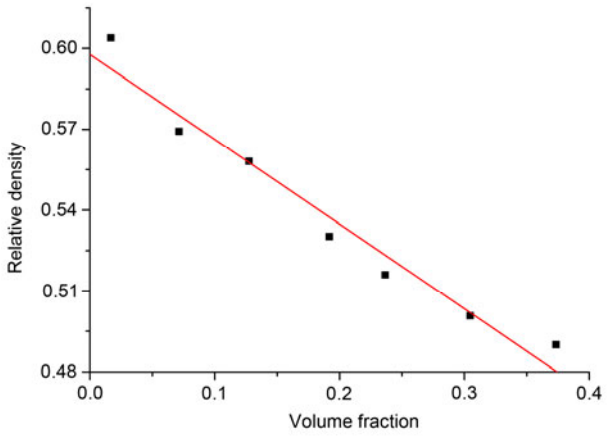

Figure 2 (Color online) The initial relative density of compacts as a function of the volume fraction of agglomerates.

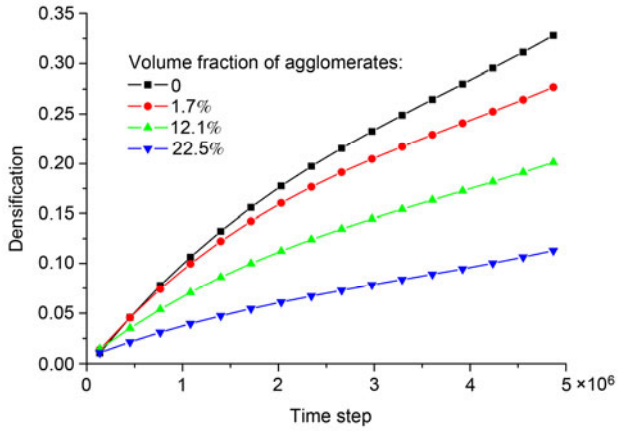

Figure 3 (Color online) The densification as a function of sintering time for systems with agglomerates with different volume fractions.

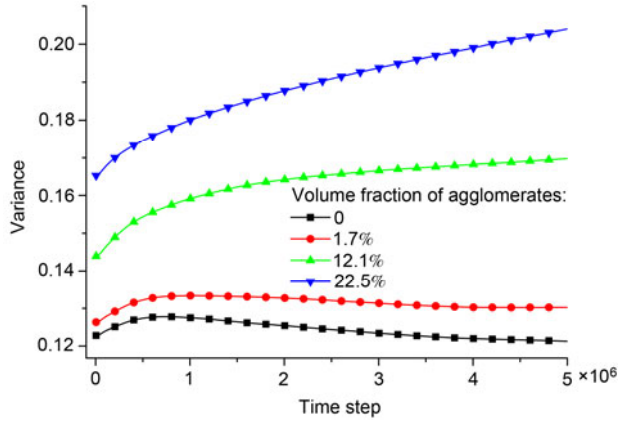

Figure 4 (Color online) The "variance" as a function of sintering time for systems with agglomerates with different volume fractions.

Figure 4. The microstructural uniformity is characterized by the parameter "variance" defined in sect. 2.2. The larger the value of "variance", the more non-uniform the microstructure is. From Figure 4, one can see that the "variance" at the initial time is larger for the compact with a larger volume fraction of agglomerates, which indicates more non-uniformity of the microstructure induced by the large volume fraction of agglomerates. As the sintering time goes, the "variance" increases consistently for compacts with more numbers of agglomerates, such as the cases with volume fractions 12.1% and 22.5%, while it increases at the early stage, and then decreases later for compacts with a relative low volume fraction of agglomerates, for example, 0 and 1.7%. One conclusion can be drawn that more agglomerates can not only induce a more non-uniform distribution of particles in the initial packing but also induce a more non-uniform microstructure during sintering.

3.2 Size effect of agglomerates

Figure 5 shows the size effect of agglomerate on the densification of particle bodies during sintering for a fixed volume fraction of agglomerates 10.2%, in which ten No. 10, five No. 11 and two No. 12 agglomerates are included, respectively. All the three types of agglomerates are spherical and the radii non-dimensioned by the radius of the particle R_{agg}/R_{par} are 2.8, 3.8 and 5.1, respectively. At the same sintering time step, the densification increases with an increasing radius of agglomerates, which is consistent with the experimental findings in Tuan et al. [19]. For a fixed volume fraction of agglomerates, the larger the size, the smaller the number of agglomerate is. As a result, densification can be improved in the case with a small number of agglomerates.

Size effect of agglomerates on the microstructural evolution of sintering bodies is also investigated with a given volume fraction of agglomerates as shown in Figure 6. The "variance" for a sintering body including more and smaller agglomerates increases more rapidly than that with less and larger agglomerates, which indicates the microstructure of a compact with more and smaller agglomerates will become more inhomogeneous during sintering.

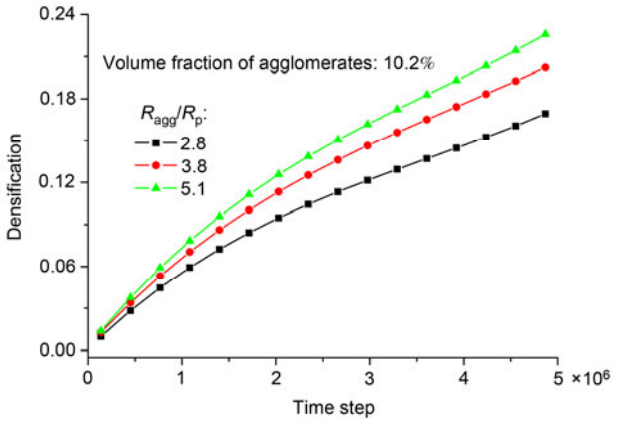

Figure 5 (Color online) The densification as a function of sintering time for systems with spherical agglomerates with a different radius but the same volume fraction.

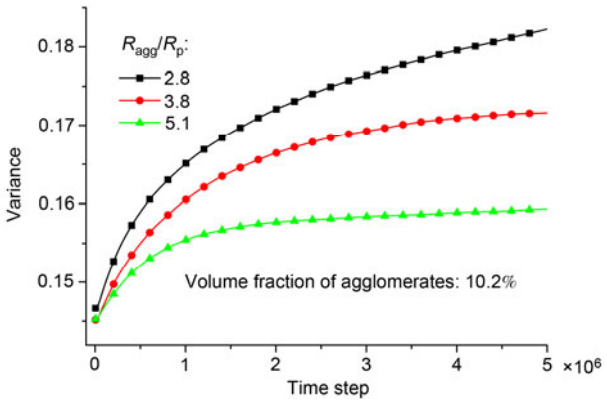

Figure 6 (Color online) The "variance" as a function of sintering time for systems with spherical agglomerates with a different radius but the same volume fraction.

3.3 Shape effect of agglomerates

Figure 7 shows the shape effect of agglomerates on the densification of sintering bodies, in which three sets of compacts are investigated. Each set consists of two kinds of compacts combined by the same uniform particles and agglomerates with the same volume fraction, the same size and the same relative density but with two kinds of different shapes. One is spherical and the other is columnar. From Figure 7, for each set of compacts, the shape of agglomerate has a nearly negligible effect on the sintering densification, especially for the cases with a small volume fraction of agglomerates.

The effect of shape of agglomerates on the microstructural evolution is shown in Figure 8. The "variance" of the system containing spherical agglomerates is a little larger than that containing columnar ones in all three sets of particle systems, which indicates that spherical agglomerate could induce a more non-uniform microstructure compared with the columnar one.

3.4 Effect of the distribution of agglomerates

The effect of the distribution of agglomerates on the sintering densification is shown in Figure 9, in which three cases are considered and each case is produced with random distribution of ten No. 1 agglomerates but with different distribution forms. From Figure 9, the distribution of agglomerates has negligible effect on the densification of the system. However, it does influence the microstructural uniformity as shown in Figure 10, which gives the "variance" as a function of sintering time step for the three cases. The "variance" of case 2 is a little larger than those of the other two ones. However, the difference in microstructural uniformity is so small that it does not reflect the densification behavior of the whole sintering system.

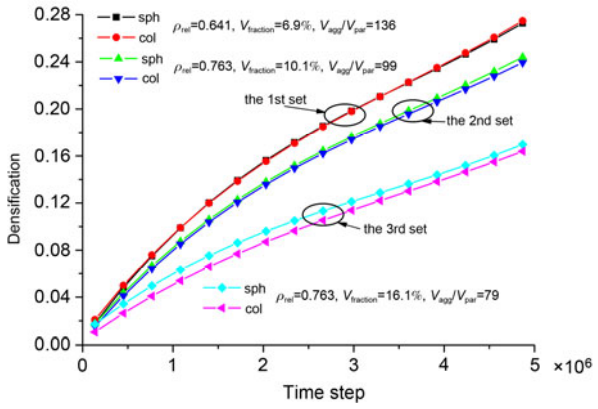

Figure 7 (Color online) The densification as a function of sintering time for systems with agglomerates with a different morphology but the same volume fraction.

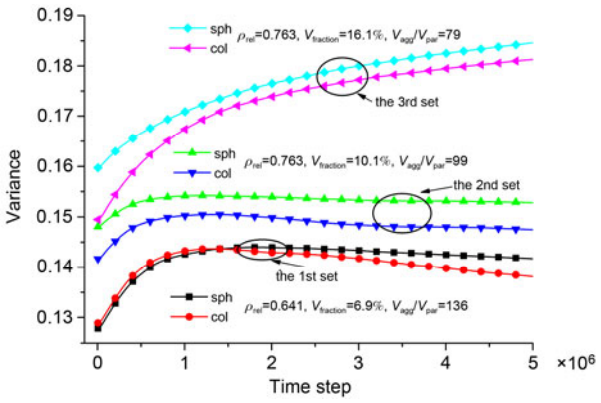

Figure 8 (Color online) The "variance" as a function of sintering time for systems with agglomerates with a different morphology but the same volume fraction.

3.5 Effect of the average coordination number of agglomerates

Figure 11 shows the effect of the average coordination number of agglomerates on the densification, in which three systems containing three No. 2 agglomerates are investigated with the same distribution form but with different average coordination numbers. The average coordination numbers of agglomerates in the three cases are 32.7, 16.0 and 5.0, respectively, which can be produced by tuning the parameter D_c to be 1.5, 1.7 and 1.9, respectively, during the

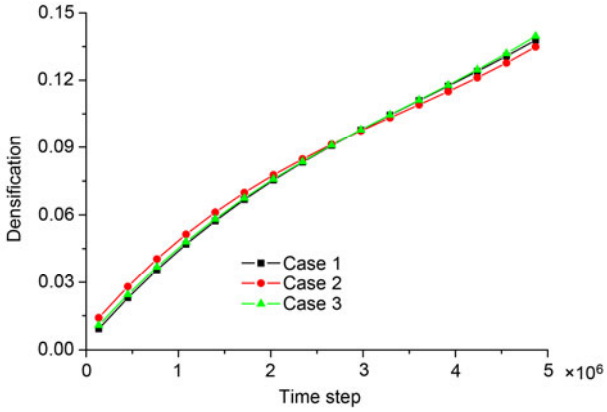

Figure 9 (Color online) The densification as a function of sintering time for systems with agglomerates with different distribution forms.

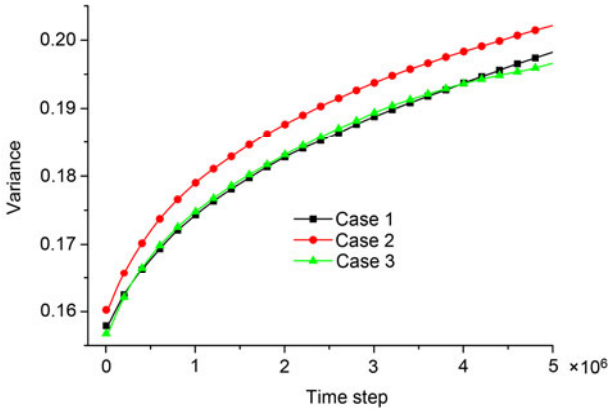

Figure 10 (Color online) The "variance" as a function of sintering time for systems with agglomerates with different distribution forms.

generation of initial numerical model. As shown in Figure 11, the densification increases with an increasing average coordination number of agglomerates at the same sintering time step. In fact, the initial densities of three systems are different, which is caused also by the initially average coordination number. The larger the average coordination number of agglomerates, the larger the initial density of a system is.

Figure 12 gives the "variance" as a function of sintering time step for cases with different average coordination number of agglomerates. The "variance" of the case with a large average coordination number of agglomerates, e.g., 32.7, increases only at the beginning of sintering simulation and then decreases. However, the "variance" of the case with a small average coordination number, e.g., 5.0, increases in the whole sintering simulation. This indicates that the agglomerates with a large average coordination number would induce a more uniform microstructure while the ones with a small average coordination number would make the microstructure more inhomogeneous. As the average coordination number increases, agglomerates contact the surrounding particles more closely and the isolated vacancies around agglomerates would be hindered to become big pores. Therefore, the microstructure of the system with a high average coordination number of agglomerates becomes more and more uniform.

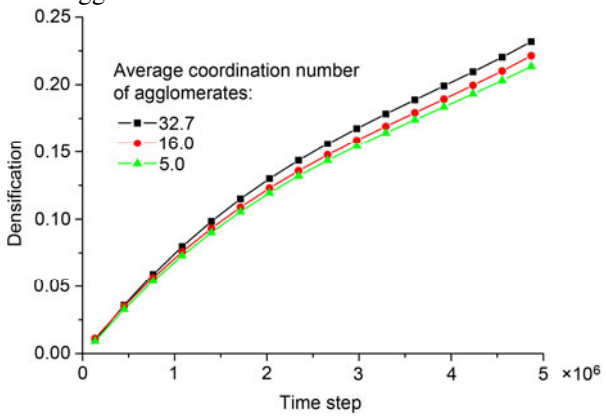

Figure 11 (Color online) The densification as a function of sintering time for systems with agglomerates with different average coordination numbers.

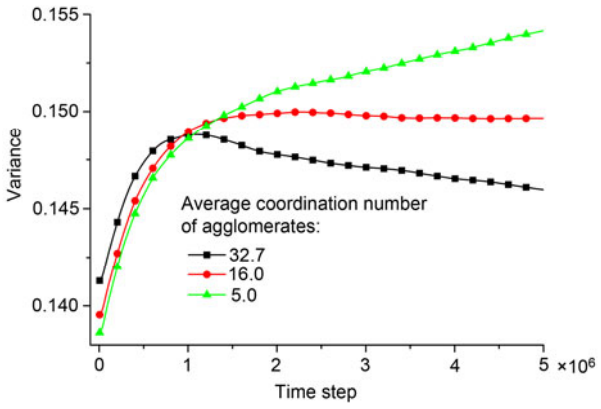

Figure 12 (Color online) The "variance" as a function of sintering time for systems with agglomerates with different average coordination numbers.

4 Conclusions

Effects of agglomerates on the densification behavior and microstructural evolution during solid-state sintering of a cube of copper particles have been studied by discrete element method in the present paper. The volume fraction, morphology, size, distribution and average coordination number of agglomerates are considered. It is found that the densification of the sintering body decreases when the volume fraction of agglomerates increases. More agglomerates will induce a more non-uniform microstructure. With a given volume fraction of agglomerates, the compact with more and smaller agglomerates has a poorer densification and more inhomogeneous microstructure. The spherical agglomerates make the microstructure a little more non-uniform than the columnar ones, but with negligible difference on the densification of the sintering bodies. The distribution of agglomerates has a negligible effect on the densification but with a minor effect on the microstructural uniformity. Agglomerates with a small average coordination number would weaken the densification rate and lead to a non-uniform microstructure.

This work is just a beginning using DEM to study the effect of various inhomogeneities on the densification behavior and microstructural evolution during solid-state sintering. A number of significant problems are still open. For example, what is the effect of rearrangement of agglomerates on the densification and microstructural evolution? What is the effect of sintering between agglomerates coupled with sintering between particles, i.e., sintering of fractal agglomerated structure [2], on the densification and microstructural evolution? Solving all these important problems would be helpful for understanding the very complex sintering process and designing sintering products with novel material properties.

The work was supported by the National Natural Science Foundation of China (Grant Nos. 10972220, 11021262 and 11125211) and the National Basic Research Program of China (Grant No. 2012CB937500).

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