Investigation on compaction behaviors of Ag₃₅Cu₃₂Zn₃₃ mixed metal powders under cold die compaction

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Abstract

Series experiments were performed to investigate the cold die compaction behaviors of Ag₃₅Cu₃₂Zn₃₃ mixed metal powders to manufacture thin sheet of cadmium-free silver based filler metal, and the effect of friction on powder compaction behaviors were analyzed using the finite element method (FEM). The density dependent modified Drucker-Prager Cap model was established to characterize the mixed metal powders compaction behaviors. The die wall friction coefficients under different lubricated conditions were experimentally determined and were modeled by Coulomb friction model in simulations. The established constitutive model and material parameters were validated by experiments and finite element simulations of powder compaction process in ABAQUS/Standard with a user subroutine USDFLD. The compaction mechanisms of Ag₃₅Cu₃₂Zn₃₃ mixed metal powders were analyzed with Gerdemann-Jablonski compaction equation, and the nonlinear compaction equation considering the friction effect was established and validated to characterize the powder compaction mechanisms under different die wall friction. With the friction coefficient increased, the relative fractional contributions of the particle rearrangement mechanisms increased and the powder deformation mechanisms contributions decreased. Finally, the influences of die wall friction on powder compaction behaviors were discussed. The results shown that the die wall friction significantly influence the powder flow behaviors and residual elasticity, which induce inhomogeneous relative density and stress distribution, cracks and capping in powder compact.

Keywords : Powder compaction, Compaction behavior, Mixed metal powders, Friction, Finite element modeling

1. Introduction

Cold die compaction is the most common method in powder metallurgy (PM) to economically fabricate complex shape metallic parts of high strength. So far, the research interest on compaction behavior of metal powder is mainly concentrated on pure powder which is attributed to the complexity of mixed powders compaction (Kim and Cho, 2001). However, various industrial processes involve the compaction of mixed metal powders and the compaction mechanisms of which were continually investigated (Bouvard, 2000, Cho and Kim, 2001, Kim et al., 2003, Hafizpour et al., 2010, Moreno et al., 2011). Recently, a process method combines powder compaction and sinter was employed to manufacture thin sheet of the brittle cadmium-free silver based filler metal which contains high proportion of Sn, Zn, In etc. additional elements (Andrieux et al., 2008, Zhou et al., 2017). In the manufacture process, multicomponent mixed metal powders of Ag, Cu, Zn and other additional elements are compressed to green powder compact, and subsequently undergoes a sinter process to attain the final thin sheets of filler metals with the desired properties. The properties of final product significantly depend on the quality of green compact, as the defects and cracks generated at powder compaction process will maintain continuously during the subsequent processes. The powder compaction process generally includes three stages, die filling, loading and unloading, ejection. The compaction densification behavior of

the mixed metal powder in the whole compaction process determines the green compact quality and directly affects the dimensional accuracy and properties of the final products. In addition the friction between the powder compact and die surface is significantly important in the powder compaction. It results in inhomogeneous stress, density distribution and powder deformation in green compact which will lead to property variations in different regions and affect the mechanical response during subsequent operations. Thus, it is of practical significance to investigate the mixed metal powders compaction behavior and the friction influence during the whole compaction process for manufacturing the cadmium-free silver based filler metals sheets. In the present work, green compacts of Ag₃₅Cu₃₂Zn₃₃ silver based filler metals were fabricated, the compaction behaviors of the multicomponent mixed metal powders under cold die compaction were investigated, and the effect of friction on the powder compaction behaviors were analyzed.

Compared with the trial and error method, the numerical modeling is an effective approach to investigate the powder compaction behavior under different friction conditions considering the efficiency and cost. Generally, the finite element method (FEM) (Lee and Kim, 2002, Chtourou et al., 2002) with continuum mechanics and discrete element method (DEM) (Jerier et al., 2011, Güner et al., 2015) were used to model the powder compaction behaviors. Individual particles were modeled in the DEM and the analysis of particles contact interaction and deformation could be realized. But the DEM is only appropriate for modeling the compaction behavior of limited number of particles at low density stage. Compared with DEM, the FEM treats the powder particles in large volume as a continuous media with a certain relative density, which is more suitable for engineering application. Based on finite element simulations, the powder compaction behaviors under different conditions are analyzed, the stress, density and powder deformation distributions could be predicted. In addition, the FEM assists to understand the influences of friction and process parameters on the powder compaction behavior, so as to realize the process parameters and tooling design optimization. However, an accurate constitutive model is the key to simulate the powder compaction. Generally, the “classical elastoplasticity” models, which were expanded from the classical von-Mises model, such as Kuhn and Downey (1971), Green (1972), Shima-Oyane (1976) models, were frequently employed to characterize the metal powder compaction behavior. While, these models were generally proposed from uniaxial compression test of sintered powder compact to model the pure metal powder compaction behavior. Moreover, these models fail to describe the shear phenomenon in powder compact which is significantly important in unloading and ejection process and are not suitable for describing the early stage of compaction (Lee and Kim, 2002, 2007). Thus a proper constitutive model is vital for characterizing the compaction behavior of Ag₃₅Cu₃₂Zn₃₃ mixed powders. To model the compaction behaviors of copper and tool steel mixed powders, mixed yield functions were proposed by Kim (Kim and Cho, 2001, Cho and Kim, 2001) with various mixing rules. Han et al. (2015) used the Voigt mixed-mode model to determine the yield strength and elastic parameters of the Fe and Al composite powders in simulations of the single action die compaction process. However, these models are too complicated and the material parameters are not convenient to obtain which are not applicable for multicomponent mixed metal powders in the present work. Since the compaction behaviors of soil materials and mixed metal powder have many similarities, such as both of them are mixtures of particles with different mechanical features, material properties varied with densities. Meanwhile, the “soil mechanics” model, modified Drucker-Prager Cap (DPC) plasticity model was frequently employed to model the metal powder compaction (Shang et al., 2012, Zadeh et al., 2013, Zhou et al., 2013). Thus, the modified DPC model was employed to characterize the compaction behaviors of Ag₃₅Cu₃₂Zn₃₃ mixed metal powder. In addition, the compaction equation considers the powder compact relative density as a linear or nonlinear function of the applied pressure which was frequently used to explain the powder behavior through theoretical considerations. It explicates the powder compaction mechanisms, such as particles slippage, rearrangement, plastic deformation and work hardening. Different linear and nonlinear compaction equations have been proposed to characterize powder compaction behaviors (Heckel, 1961, Cooper and Eaton, 1962, Kawakita and Lüdde, 1971, Ge, 1991, Gerdemann and Jablonski, 2011). The compaction behaviors of mixed metal powders or metal matrix composite powders were investigated with the proposed compaction equations (Hesabi et al., 2007, Sivasankaran et al., 2011, Ghiță and Popescu, 2012), in which the compression properties and compaction mechanisms of mixed powders were analyzed. Meanwhile, the effect of friction or lubrication on the metal powder compaction behavior were analyzed and evaluated by compaction equations (Enneti et al., 2013, Lou et al., 2015). Thus the finite element modeling combines with the compaction equation method can comprehensively characterize the powder densification behaviors under different friction conditions and the influence of friction on powder compaction behaviors could be analyzed and evaluated.

In this work, green compacts of Ag₃₅Cu₃₂Zn₃₃ cadmium-free silver based filler metals were fabricated, the

compaction behaviors of the multicomponent mixed metal powder were investigated and the influences of die wall friction were analyzed. The density dependent modified DPC plasticity model was employed to model the Ag35Cu32Zn33 mixed powders compaction behavior, and all the material parameters were experimentally calibrated. Validation experiments and finite element simulations of powder compaction process with a user subroutine USDFLD in ABAQUS/Standard were performed and compared to verify the established material model. Based on simulations, the effect of friction on powder compaction mechanisms were evaluated with a nonlinear compaction equation and the influence of die wall friction on Ag35Cu32Zn33 mixed powders compaction behaviors were discussed. The findings presented in the present work are expected to provide a fundamental understanding of the influence of friction on compaction behavior of multicomponent mixed metal powder, which can be used in tool design and process parameters optimization to manufacture thin sheets of cadmium-free Ag35Cu32Zn33 filler metals.

2. Constitutive model and compaction equation

2.1 Modified Drucker-Prager Cap constitutive model

The modified Drucker-Prager Cap (Han et al., 2008) model is a continuum mechanics model which assumes that the material is isotropic. The model contains three yield surfaces as shown in Fig. 1. The shear yield surface F_s provides a shearing flow criterion, the cap yield surface F_c is an elliptical curve which provides inelastic hardening mechanisms or controls volume dilatancy to represent plastic compaction of material or material yields in shear. And the transition surface F_t is defined to facilitate the numerical simulation. The yield surfaces and plastic flow potential are defined with the hydrostatic pressure stress p and Mises equivalent stress q . The equations of the three surfaces are expressed as

$$F_s = q - p \tan \beta - d = 0 \quad (1)$$

$$F_c = \sqrt{(p - p_a)^2 + \left[\frac{Rq}{1 + \alpha - \alpha/\cos \beta} \right]^2} - R(d + p_a \tan \beta) = 0 \quad (2)$$

$$F_t = \sqrt{(p - p_a)^2 + \left[q - \left(1 - \frac{\alpha}{\cos \beta} \right) (d + p_a \tan \beta) \right]^2} - \alpha(d + p_a \tan \beta) = 0 \quad (3)$$

where d and β represent the cohesion and friction angle which determine the shear yield surface F_s . R and α are parameters controlling the shape of the cap yield surface and the transition yield surface. Typically, α is a constant value between 0.01 and 0.05, in the present work α is given by 0.02. p_a and p_b are evolution parameter and hydrostatic compression yield stress, respectively. p_b is usually expressed as

$$p_b = f(\varepsilon_v^{pl}) \quad (4)$$

where ε_v^{pl} is the volumetric plastic strain and is defined as

$$\varepsilon_v^{pl} = \ln(\rho/\rho_0) \quad (5)$$

where ρ and ρ_0 represent the current relative density of powder compact and initial filling relative density, respectively. The relative density ρ is determined as the powder compact current density divide by the theoretical density of mixed powders. The theoretical density of mixed powder is determined according to the mass fraction of each pure powder.

Fig. 1. The modified Drucker-Prager Cap model yield surfaces (Han et al., 2008).

To fully define the DPC model, five parameters d , β , p_a , R , and p_b are required. Meanwhile, the Young's modulus E and Poisson ratio ν are required to characterize the powder elastic properties. And friction coefficient μ is also need to describe the friction condition between powder compact and die. To determine all the model parameters, series experiment procedures including uniaxial tension, pure shearing, diametrical compression, uniaxial compression and powder instrumented die compaction tests (as shown in Fig. 2) could be used, more details about the calibration method could be found in elsewhere (Han et al., 2008). In this paper, uniaxial compression and diametrical compression tests were carried out to determine d and β which were calculated as

$$d = \frac{(\sqrt{13}-2)\sigma_c\sigma_t}{\sigma_c-2\sigma_t} \quad (6)$$

$$\beta = \tan^{-1} \left[\frac{3(\sigma_c-d)}{\sigma_c} \right] \quad (7)$$

where σ_c and σ_t are the axial compressive and radial tensile strength measured by uniaxial compression and diametrical compression tests, respectively.

The cap surface parameters, R , p_a , p_b were calibrated by analyzing the stress state on the intersection point B(p_0, q_0) between the loading path and the cap surface using the powder instrumented die compaction test (Han et al., 2008). The Young's modulus E and Poisson ratio ν were determined with a linear elasticity law from the unloading data in the powder die compaction tests.

$$R = \sqrt{\frac{2(1+\alpha-\alpha/\cos\beta)^2}{3q_0}}(p_0-p_a) \quad (8)$$

$$p_a = \frac{-3q_0+4d\tan\beta(1+\alpha-\alpha/\cos\beta)^2+\sqrt{9q_0^2+24dq_0(1+\alpha-\alpha/\cos\beta)^2\tan\beta+8(3p_0q_0+2q_0^2)[(1+\alpha-\alpha/\cos\beta)\tan\beta]^2}}{4[(1+\alpha-\alpha/\cos\beta)\tan\beta]^2} \quad (9)$$

$$p_b = p_a(1+R\tan\beta)+Rd \quad (10)$$

$$\frac{d\sigma_r}{d\sigma_z} = \frac{\nu}{1-\nu} \quad (11)$$

$$\frac{d\sigma_z}{d\varepsilon_z} = \frac{(1-\nu)E}{(1-2\nu)(1+\nu)} \quad (12)$$

where $d\sigma_r$, $d\sigma_z$ and $d\varepsilon_z$ represent the increments of the radial stress, axial stress and axial strain, respectively.

Fig. 2. The DPC model parameters identification: (1) uniaxial tension, (2) pure shear, (3) diametrical compression, (4) uniaxial compression, and (5) powder instrumented die compaction tests.

2.2 Compaction equation

The compaction equations present relationship between powder relative density and applied pressure, which were widely used to describe the powder compaction behavior. It stems from the practical requirement of accurately predicting the compaction pressures needed to compress the powder to a certain density. To exactly evaluate the role of powder rearrangement and powder deformation mechanisms to powder densification during powder compaction under different friction conditions, the nonlinear Gerdemann-Jablonski (Gerdemann and Jablonski, 2011) compaction equation was employed to analyze the effect of friction on compaction mechanisms. The Gerdemann-Jablonski equation considers three mechanisms, an initial density and two independent pressure terms which represent powder particle rearrangement mechanism (including the particles sliding and fracture) and work hardening mechanisms (the sum of the particles elastic and plastic deformation). The two-phase exponential equation was expressed as

$$D = D_0 + A(1 - e^{-aP}) + B(1 - e^{-bP}) \quad (13)$$

where D_0 is the powder initial relative density. A and B are parameters reflect the relative contributions of powder rearrangement and work hardening to powder densification, respectively. $\ln 2/a$ and $\ln 2/b$ represent the process half-lives of the powder rearrangement mechanism and work hardening mechanism according to the interpretation proposed by Machaka (Machaka and Chikwanda, 2015).

3. Material and experiments

3.1 Material

The compositions and physical property of Ag35Cu32Zn33 mixed powders were shown in Table 1. To prepare Ag35Cu32Zn33 mixed metal powders, pure metal powders were weighed by an electronic balance according to their mass fractions and were dried in the oven for one hour. Subsequently, the powders were homogeneously mixed by ball milling. The powder morphology was illustrated by SEM images as shown in Fig. 3. The theoretical density of Ag35Cu32Zn33 mixed powders is calculated according to the mass fraction of each pure powder as a value of 8.676 g/cm³.

Table 1. The compositions and physical property of Ag35Cu32Zn33 mixed metal powders.

Mixed metal Powders	Compositions	Mass fractions (wt%)	Density (g/cm ³)	Particle size (μm)	Theoretical density (g/cm ³)
Ag35Cu32Zn33	Ag	35	10.53	1~45	8.676
	Cu	32	8.94	1~38	
	Zn	33	7.14	1~38

Fig. 3. The morphology of Ag35Cu32Zn33 mixed powders.

3.2 Experiments

To investigate the compaction behaviors of Ag35Cu32Zn33 mixed powders and determine the model parameters, series of diametrical compaction, uniaxial compaction and powder instrumented die compaction tests were performed by using a WAW-600 (600 KN, HUALONG, China) material testing system. Uniaxial compaction and diametrical compaction tests as shown in Fig. 2 were carried out on samples of different density levels to measure the axial compressive strength σ_c and radial tensile strength σ_t of compacts. In uniaxial compaction test, the specimen diameter and height are 10 mm and 15 mm, respectively. The compaction speed is 2 mm/min, and the maximal axial compression force F_{com} was recorded, from which the axial compressive strength was determined as $\sigma_c = 4F_{com} / \pi D^2$. In diametrical compaction test, the specimens were compressed along the diameter direction with a compaction speed of 0.25 mm/min as shown in Fig. 2. During the diametrical compaction, the tensile failure initiated at the specimen center and the green compacts crushed into two halves (as shown in Fig. 4), the specimen thickness to diameter aspect ratio t/D should be ≤ 0.25 (Shang et al., 2012). The specimen diameter is 16 mm, the specimen thickness is ≤ 4 mm in all diametrical compaction tests. The crushing force F_{ten} was determined during the diametrical compaction test, and the radial tensile strength was then deduced as $\sigma_t = 2F_{ten} / \pi Dt$.

Fig. 4. The specimens broken in diametrical compression tests.

The powder instrumented die compaction tests were performed with the compaction apparatus as shown in Fig. 5. The internal diameter, external diameter and height of the die are 10 mm, 20 mm and 50 mm, respectively. In the instrumented die compaction tests, an amount of 4.77 g Ag35Cu32Zn33 mixed powder was manually poured in the die to achieve a uniform packing. The initial filling density was determined from the measured powder filling height, the initial relative density of powder filling in die compaction was calibrated as 0.482. During loading and unloading process, the top punch moved downward and upward with a constant load rate 0.2 KN/s to press the powder to different relative densities level, and the bottom punch kept stationary. The strain-gauge measurement method (Zhou et al., 2017) was employed to measure the radial stress during the powder compaction. The load cylinder displacement, the axial compaction forces of top/bottom punches and radial stresses between powder compact and die were measured in the instrumented die compaction tests. Two experiment cases are designed to characterize the differences of the friction

between powder compact and die under different lubricant conditions. Die compaction tests in an unlubricated die, the inner die surface was cleaned by acetone and ethanol. Die compaction tests in lubricated die, the inner die surface was lubricated with zinc stearate alcohol solution to reduce the friction during the consolidation process.

Fig. 5. The experimental apparatus used in powder die compaction test.

4. Result and discussion

4.1 Determination of the model parameters

The axial compressive and radial tensile strengths of Ag35Cu32Zn33 powder compacts were measured by series of uniaxial compression and diametrical compression tests and were shown in Fig. 6. The parameters d and β were then determined by Eqs. (6) and (7) as shown in Fig. 7, and the experimental data were fitted as functions of relative density.

Fig. 6. Strengths of Ag35Cu32Zn33 powder compacts varied with relative density.

Fig. 7. Shear failure surface parameters of Ag35Cu32Zn33 mixed powders: (a) d , (b) β .

In the powder die compaction test, the axial stress was determined as the top punch force divided by the powder

compact cross section area. And the axial strain was determined with the change of powder height divided by the powder initial filling height. Thus the axial stress-axial strain and radial stress-axial strain curves of powder compacts with different relative density were determined in die compaction tests as presented in Fig. 8. Then the cap surface parameters R , p_a , p_b were calculated by analyzing the stress state on the intersection point between the loading path and the cap surface as described in section 2.1. R and p_a were determined and varied with relative density, p_b is determined and related to the volumetric plastic strains ε_v^{pl} . All the cap failure surface parameters were fitted as shown in Fig. 9. The Young's modulus E and Poisson ratio ν were determined from the unloading data in powder die compaction tests with Eqs. (11) and (12) as shown in Fig. 10. All the calculated plasticity and elastic model parameters were precisely fitted with high R^2 values, from which the model parameter in initial compaction stage (relative density < 0.62) were extrapolated and used in finite element modeling.

Fig. 8. The experiment curves of die compaction tests: (a) axial stress-axial strain curves, (b) radial stress-axial strain curves.

Fig. 9. Cap failure surface parameters of Ag35Cu32Zn33 mixed powders: (a) R , (b) p_a , (c) p_b .

Fig. 10. Elastic parameters of Ag35Cu32Zn33 mixed powders: (a) E , (b) ν .

4.2 Friction coefficient between the powder compact and die

The friction between the powder compact and die is very important for powder compaction, as it can lead to property variations and defects in powder compact. So it is significant to accurately determine the friction coefficient μ . In the present work, μ is determined with die compaction test using the Janssen-Walker theory (Sinka et al., 2003).

$$\mu = \frac{D\sigma_B}{4H\sigma_r} \left(\frac{\sigma_T}{\sigma_B} \right)^{\frac{Z}{H}} \ln \frac{\sigma_T}{\sigma_B} \quad (14)$$

where H and D are the powder compact height and diameter, respectively. σ_B , σ_T are the axial pressures applied on the bottom and top punches measured in the powder instrumented die compaction tests, σ_r is radial stress at the measured point which has a distance of Z from the powder compact bottom surface.

Instrumented die compaction tests were performed for Ag35Cu32Zn33 mixed metal powders using lubricated/unlubricated die. The friction coefficients μ in lubricated and unlubricated die were determined and varied with the contact pressure as shown in Fig. 11. It can be observed that for both two cases, the friction coefficient increased sharply with the increase of contact pressure at low pressure region (≤ 20 MPa), and then decreased gradually. But at the high pressure region (> 40 MPa), μ varied not obviously and almost maintained a constant value, 0.09 for lubricated die and 0.19 for unlubricated die. It indicates that the lubrication (Zinc stearate alcohol solution) has great influence on the die wall friction.

Fig. 11. The friction coefficients measured in powder die compaction tests.

4.3 Validation of the model parameters

To validate the determined plasticity and elastic model parameters, validation experiments of Ag35Cu32Zn33 powder die compaction tests were performed in lubricated/unlubricated die. Powder compacts were produced by die compaction test with lubricated and unlubricated die, and the characteristics of the two compacts in the validation

experiments, such as initial filling height H_0 , the top punch displacement H_1 , initial filling relative density ρ_0 , final average relative density ρ_1 and compaction pressure P , are presented in Table 2. Moreover, finite element simulations of the powder die compaction process in validation experiments for lubricated/unlubricated die were performed, the experimental and simulated results were compared to verify the material parameters. As the geometry corresponding to the die compaction process was axisymmetric, thus a quarter of powder compaction model was established in ABAQUS/Standard as shown in Fig. 12. In simulations, a cylindrical powder compact with a diameter of 10mm was treated as deformable continuum and meshed with solid elements C3D8R. The die and punches were set as rigid bodies. The inner and outer diameters of the die are 10mm and 20mm, respectively. The powder initial relative density, initial height and the top punch displacement were set according to validation experiments as shown in Table 2. The boundary condition between the powder and tool surface was modeled as a friction surface interaction by the Coulomb friction model using penalty function. As shown in Fig. 11, μ almost maintained a constant value at the high pressure region ($> 40\text{MPa}$), so the friction coefficient at interface between the powder and die, punches were defined as a constant value (Table 2) according to the experimental measurements. The experimentally calculated plasticity and elastic model parameters in section 4.1 were implemented in ABAQUS to simulate the cold die compaction process of Ag35Cu32Zn33 mixed powders. As all the model parameters varied with the relative density, the user subroutine USDFLD which defines a field variable of relative density in ABAQUS was used to characterize the evolution of model parameters with the relative density. From which the plasticity and elastic material parameters during each time step were updated before calculation. The simulations of powder compaction were modeled as quasi-static, the geometry and loading sequence (single-ended compaction) were designed according to the validation experiments in powder die compaction test.

Table 2. The characteristics of powder compact in the validation experiments.

Compacts	m (g)	H_0 (mm)	H_1 (mm)	ρ_0	ρ_1 (experiment)	ρ_1 (simulation)	P (MPa) (experiment)	P (MPa) (simulation)
Lubricated die $\mu=0.09$	4.788	14.46	6.88	0.486	0.908	0.914	389.2	398.1
Unlubricated die $\mu=0.19$	4.767	14.54	6.79	0.481	0.892	0.901	395.9	405.7

Fig. 12. Finite element modeling of powder compaction.

The simulated results of relative density distributions in powder compacts after decompression for lubricated and unlubricated die were presented in Fig. 13, and from which the final average relative density of powder compacts after decompression were calculated. The comparisons between the simulated and experiment results were shown in Fig. 14. The simulated curves all matched well with the experimental data both for lubricated die ($\mu=0.09$) and unlubricated die ($\mu=0.19$), and the simulated results of the final relative density and maximum compaction pressures were almost consist with the experimental measurements as presented in Table 2. The good agreement indicates that the determined model parameters are able to precisely characterize the compaction behaviors of Ag35Cu32Zn33 mixed powders, and could be further used for process parameters optimization simulation.

Fig. 13. Relative density distributions in powder compacts after decompression: (a) lubricated die $\mu=0.09$, (b) unlubricated die $\mu=0.19$.

Fig. 14. Comparisons between the simulated and experiment results.

4.4 Effect of friction on Ag35Cu32Zn33 mixed powders compaction mechanisms

Density-pressure curve was widely used to characterize the powder compaction behavior, from which the compaction mechanisms can be analyzed by compaction equation as describe in section 2.2. Simulations of powder compaction under different die wall friction conditions were carried out, the relationship of relative density and compaction pressure under different friction coefficients were calibrated. The calibrated data were fitted and analyzed using the nonlinear Gerdemann-Jablonski compaction equation (Eq. 13) as shown in Fig. 15, and the fit parameters were list in Table 3. It was found that the nonlinear equation characterizes well the powder compaction behavior over the entire pressure range which is confirmed with the high R^2 values. As shown in Table 3, the fit values of D_0 were almost consist with the experimental measured value (0.472 ± 0.01) which indicates that the nonlinear Gerdemann-Jablonski compaction equation describes well the mixed metal powder compaction mechanisms at the initial compaction stage under low pressures. The fit values of A increased with the friction coefficients which represent the relative contributions of powder rearrangement to powder densification increased. While the fit values of B almost decreased with the increase of friction coefficients, it means that the relative contributions of powder deformation mechanism in powder compaction decreased with the increase of the friction coefficients. In addition, the values of $\ln 2/a$ and $\ln 2/b$ significantly increased with the increase of friction coefficients as presented in Table 3, which indicates the particle rearrangement and powder deformation over a wider range of compaction pressure and the process half-lives of them become longer when the friction coefficients increased. With the fitted parameters tabulated in Table 3, the relative fractional contribution of the powder rearrangement and work hardening (powder deformation) mechanisms to Ag35Cu32Zn33 mixed metal powders densification were deduced and shown in Table 4. It is shown that the friction has a significant effect on the compaction mechanisms of Ag35Cu32Zn33 mixed metal powders. With the friction coefficient increased, the relative fractional contribution of the powder rearrangement mechanism increased and the powder deformation mechanism contribution decreased. These phenomena were attributed to the increased die-wall friction reduced the pressure transferred to the powder bed and obstructed the

powder deformation, which caused the decrease of apparent compressibility of the powder and increased the process half-lives of the particle rearrangement and powder deformation. Meanwhile, the relative contributions of particle rearrangement and powder deformation mechanisms to powder densification during compaction process changed with the increased die-wall friction. The relationship between Gerdemann-Jablonski equation parameters and friction coefficient μ were determined as shown in Table 3, and then the nonlinear compaction equation considering the friction effect was deduced. To validate the determined equations, the experimental data of lubricated and unlubricated die were compared with the calculated results as presented in Fig. 16. The good agreement between the experiment data and calculated result indicates that the determined functions of Gerdemann-Jablonski equation parameters with friction coefficient could characterize the density-compaction pressure relationship for the Ag35Cu32Zn33 mixed metal powders under different die wall friction, from which the powder compaction mechanisms could be further analyzed.

Fig. 15. The relative density and compaction pressure data fitted with the Gerdemann-Jablonski equation.

Table 3. Fit Parameters of the Gerdemann-Jablonski equation (Eq. 13).

μ	D_0	A	B	a	b	$\ln 2/a$ (MPa)	$\ln 2/b$ (MPa)	R^2
0	0.4737	0.1189	0.3842	0.0409	0.0052	16.9474	133.2975	0.99994
0.1	0.4686	0.1373	0.3789	0.0325	0.0044	21.3276	157.5335	0.99989
0.2	0.4681	0.1678	0.37	0.0235	0.0033	29.4956	210.0446	0.99959
0.3	0.4686	0.1974	0.3696	0.0183	0.0024	37.8769	288.8113	0.99936
0.4	0.4676	0.2202	0.3806	0.0151	0.0018	45.9038	385.0818	0.99922

$$A=0.2627\mu+0.1158; R^2 = 0.9941$$

$$B=1.25\mu^3-0.4564\mu^2-0.0264\mu+0.3843; R^2 = 0.9993$$

$$a=0.0408e^{-2.567\mu}; R^2 = 0.9934$$

$$b=0.0088\mu+0.0052; R^2 = 0.9934$$

Table 4. Relative fractional contributions of powder rearrangement and work hardening mechanisms to the final density achieved.

μ	Powder Rearrangement (%)	Work Hardening (%)
0	23.63	76.37
0.1	26.60	73.40
0.2	31.20	68.80
0.3	34.81	65.19
0.4	36.65	63.34

Fig. 16. Validation of the compaction equation contain friction coefficient.

4.5 Effect of friction on Ag35Cu32Zn33 mixed powders compaction behaviors

The compaction process of Ag35Cu32Zn33 mixed powders under different friction condition was simulated, and the densification behaviors in powder compact were investigated. As shown in Fig. 13, the relative density at the top corner in powder compact contact with the die wall is relative higher, while at the bottom corner, the relative density is lower. And the density gradient is more significant in powder compact for unlubricated die. The Mises stress distributions in powder compact after compaction and decompression for lubricated and unlubricated die were shown in Fig. 17. Obviously, higher stress concentration occurs in the top area and the stress in the bottom corner is lower both for two friction conditions after compaction and decompression. This phenomenon is accordance with the relative density distribution. Meanwhile, due to the higher friction coefficient in unlubricated die, the stress gradient generated in powder compact is more significant. The existence of relative density and stress gradient during powder compaction is attributed to the influence of the die wall friction on powder flow behaviors during the compaction process. Two powder flow behaviors in the die, such as the axial powder flow and radial powder flow, were analyzed in the green compact of mixed metal powders. Fig. 18 shows the distributions of axial displacement in powder compact after compaction under different friction conditions, and the displacement evolution with radial distance under different friction coefficients at height of $H=1.93$ mm, 3.86 mm and 5.79 mm were analyzed and presented in Fig. 18 (c). As the powder compaction test is a single-ended compaction process, the axial pressure is transmitted from the top punch to the lower layer, thus the axial displacements in powder compact increase gradually from bottom to top, which induce that the relative density and stress in powder decrease from top surface to bottom layer. It is also clear that the existence of axial displacement variation along radial distance at different compact heights. Due to the effect of friction, the axial displacements in the regions closed to the die wall were less than the central region at each height. With the increase of friction coefficient, the axial displacement gradient along the radial distance is also more significant, and the corresponding stress and density distributions gradient is more obvious. While without die wall friction effect ($\mu=0$), the axial displacement at each compact height remains unchanged along the radial direction. It indicates that the die wall friction has a great influence on the axial powder flow during compaction, which is the key factor of causing stress and relative density gradients in powder compact.

Fig. 17. The Mises stress (MPa) distributions in powder compacts: for lubricated die $\mu=0.09$, (a) after compaction, (b) after decompression, and for unlubricated die $\mu=0.19$, (c) after compaction, (d) after decompression.

Fig. 18. The axial displacement (mm) distributions in powder compacts: (a) lubricated die $\mu=0.09$, (b) unlubricated die $\mu=0.19$, and (c) axial displacements under different friction coefficient.

The radial displacement distributions in powder compact after compaction for lubricate and unlubricated die were presented in Fig. 19. It was observed that convective powder flows appeared in the powder compact both for lubricated and unlubricated die, and induced two distinct parts of radial displacement. At the top area, the powder close to the die wall flow inward to powder center, while the powder at the bottom area flow outward from the powder center to the die wall. The convective powder flows in radial indirection make the powder in the top/bottom die wall region flow towards/outwards the center of compact, which induces the relative density and stress in the top/bottom areas close to the die wall are relative high/low compared with other region in powder compact (as shown in Fig. 13 and 17). It is also found that due to the higher die wall friction coefficient in unlubricated die, the radial displacement variation range and the convective powder flows along radial direction are more significant, which means a higher gradient of relative density and stress in powder compact.

Fig. 19. The radial displacement (mm) distributions in powder compacts: (a) lubricated die $\mu=0.09$, (b) unlubricated die $\mu=0.19$.

During the decompression process, with the compaction pressure decreased gradually, the powder compact spring back and the inhomogeneous stress concentrations form in compact which could result in cracks. The residual elasticity R_e was used to characterize the degree of spring back in powder compact and expressed as $R_e = \left[(l - l_0) / l_0 \right] * 100\%$, where l_0 and l are the powder compact heights after compaction and decompression. The axial residual elasticity at different heights in powder compact for lubricated die and different friction conditions were evaluated and presented in Fig. 20. It was found that the axial residual elasticity decreased when the powder height increased as shown in Fig. 20 (a), and the residual elasticity at each height decreased gradually along with the radial distance due to the effect of friction. Furthermore, with the friction coefficient increased, the axial residual elasticity at each height decreased, and the difference of axial residual elasticity along the radial direction is more significant. The larger the friction between powder compact and die, the more inhomogeneous the axial residual elasticity in powder compact. Due to the effect of die wall friction, the inhomogeneous spring back occurs in decompression, which could induce inhomogeneous stress distribution and cracks in powder compact. The axial stress distributions in powder compact after decompression under different friction conditions were presented in Fig. 21. Due to the inhomogeneous axial residual elasticity in powder compact (see Fig. 20), intensive stress bands running from the top surface towards the center of the compact were formed in the compact after decompression. In the stress bands both for lubricated and unlubricated die, the sign of the axial stress change from negative to positive. It means that part of powder compact was subjected to tension effect and the other part was subjected to compaction stress. And this phenomenon could induce capping and cracks in powder compact during the ejection. In addition, with the higher friction in unlubricated die, the stress range is more extensive which will increase the risk of crack creation and propagation.

Fig. 20. The axial residual elasticity at different heights in powder compact: (a) lubricated die $\mu=0.09$, (b) under different friction conditions.

Fig. 21. The axial stress (MPa) distributions in powder compacts after decompression: (a) lubricated die $\mu=0.09$, (b) unlubricated die $\mu=0.19$.

5. Conclusions

The compaction behaviors of Ag35Cu32Zn33 mixed powders under cold die compaction were investigated, and the effect of friction were analyzed by experiments and finite element modeling. The conclusions were summarized as follows:

- (1) The compaction behaviors of Ag35Cu32Zn33 mixed powders were characterized by modified Drucker-Prager cap model. The plasticity material parameters were determined by series of experiments and the elastic parameters E and ν were determined with a linear elasticity law. The determined material parameters were validated by experiments and finite element simulations of powder compaction process in ABAQUS/Standard through the user subroutines USDFLD. Good agreements between the simulated and experiment results indicate that the experimentally established model can precisely model the compaction behaviors of Ag35Cu32Zn33 mixed powders which could be further used for process parameters optimization simulations.
- (2) The friction coefficients between the powder and die surface under different lubricated condition were experimentally determined using the Janssen-Walker theory. It was shown that μ varied with the contact pressure, and when the contact pressure is $> 40\text{MPa}$, μ kept a constant value, 0.09 for lubricated die and 0.19 for unlubricated die. In simulations, the friction condition at interface between powder compact and punches, die was characterized by Coulomb friction model with a constant value according to the experimental measurements.
- (3) The compaction mechanisms of Ag35Cu32Zn33 mixed powders under different friction condition were analyzed. It was found that the friction has a significant influence on the compaction mechanisms of Ag35Cu32Zn33 mixed powders. With the friction coefficient increased, the relative fractional contributions of the powder rearrangement mechanisms increased and the powder deformation mechanisms contributions decreased. Meanwhile, the nonlinear Gerdemann-Jablonski compaction equation considering the friction effect was deduced and validated by experiment data, which indicates that the determined equations could characterize the Ag35Cu32Zn33 mixed powders compaction mechanisms under different die wall friction.
- (4) The effect of friction on Ag35Cu32Zn33 mixed powders compaction behaviors was analyzed. It was found that the die wall friction significantly influence the powder flow behaviors both in axial and radial directions during compaction, which induce inhomogeneous relative density and stress distribution. In addition, the friction has great influence on the axial residual elasticity in powder compact, and intensive stress bands formed in powder compact after decompression which could induce cracks and capping during ejection. With the increase of friction, the risk of crack creation and propagation increased.

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