Effects of particulate agglomerated degree on deformation behaviors and mechanical properties of in-situ ZrB₂ nanoparticles reinforced AA6016 matrix composites by finite element modeling

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Abstract
In this paper, both the tensile deformation behaviors and mechanical properties of in situ ZrB₂ nanoparticles reinforced AA6016 matrix composites are investigated with finite element analysis. For the modeling, the volume fraction of nano ZrB₂ particles is defined by combining cluster sizes with particulate agglomerated degree which was observed in the agglomeration clustered regions by experiments. The effects of clusters on the mechanical behaviors of composites are disclosed according to qualify mechanical behaviors of local particulate agglomeration. The results indicate that finite element analysis is performed to predict the Young’s modulus of composites, which is in good agreement with the experimental results. In addition, the speed of stress concentration is faster when the agglomerated degree elevates, which minimizes elongation of composites under the same particle volume fraction. Likewise, under the same agglomerated degree, the higher particle volume fraction of composites triggers the reduction of elongation.

1. Introduction

In-situ nano-particle reinforced aluminum alloy matrix composites are applied widely in the automotive and aerospace industries, due to their low density, high specific strength and stiffness and good thermal stability [1–4]. Notably, a remarkable shortcoming, that is, the agglomeration of particle reinforcements which is characterized by the clustered regions distributing around the grain or phase boundaries, has constrained the optimal coupling of strength and plasticity of Al alloy matrix composites. In the past few years, direct melting reaction (DMR) which is a typical in situ processing method has been devoted to modify the distribution of particles in matrix and to suppress the agglomerations [5–7]. Nevertheless, the agglomeration of particles is still a challenge [8]. It is worth emphasizing that the modification of clustered regions into quasi-network distribution can improve both strength and plasticity [9–11]. On this point, improving the uniformity in the dispersion of particles and modifying the distribution of clustered regions are expected to be as preferable approaches for the enhancement of mechanical properties of Al alloy matrix composites. Toward this aim, it is valuable to disclose the relationship between clustered regions and the deformation of nano-particle reinforced aluminum alloy matrix composites.

Various numerical simulations and ab-initio initial approaches are proposed to correlate micro-structure with mechanical properties of particle reinforced metal matrix composites (PRMMCs). Among them, finite element analysis (FEA) has been demonstrated to be an effective method and various models have been developed which are contributed to elucidate the effects of particle size, morphology or distribution on elastic-plastic behaviors and damage evolution of particle-reinforced metal matrix composites under uniaxial tension. Weng et al [12] discovered that the flow strength of 15 vol% SiC/Al composites is increased with decreasing particle size and the load bearing capacity of particles is decreased simultaneously. Tan et al [13] has revealed that the applied stresses for the occurrence of debonding in the SiCp/2024Al hybrid composites were increased with the decreased particle size and suggested that the mechanical properties could be further improved by optimizing particle size.
Clearly, the reduction of particle size has been as the important consideration in the development of novel Al alloy matrix composites. Furthermore, Liu et al. [14] suggested that the particle size distribution has greater influence on the degree of stress concentration than the size itself, and as to morphology of particles, irregular particles will bear more loads in the composites than those reinforced by spherical ones. On the other hand, the uniformity in the distribution of particles is another important consideration although the nonuniform distribution of the particle size, such as bimodal distribution [15, 16], can generate the positive effects in the mechanical properties of Al alloy matrix composites.

In the previous debate, agglomeration of particles were simulated via the alteration of particle arrangement in the composites [17]. In the recent literature, representative volume element (RVE) models have been established to simulate the microstructure of PRMMCs where there are numerous clustered regions [18]. It was discovered that the incipient damage occurred in the agglomeration locations at a relatively smaller strain in the composites, suggesting that agglomeration nucleates voids and accelerates the propagation of internal cracks, causing the reduction of ductility. However, there is no clear indication for the effect of various particulate agglomerated degree on elastic-plastic deformation of in situ ZrB2/AA6016Al alloy composites. There remains a need to consider characteristics of particulate agglomeration regions.

In this study, it aims to quantify characteristics of agglomerations, which are regarded as the individual reinforced structure in composites instead of particles for investigating effect of different agglomerated ratios of clustered regions on composites. The in situ ZrB2/AA6016Al alloy composites was selected as model material, the microstructural information of both particles and agglomerations is obtained from scanning electron microscope images. With finite element models (FEMs), the relationship of particulate agglomerations and deformation behavior were discussed. An elastic-plastic deformation under different loading steps was provided for understanding deformation process and analyzing morphology of fracture surfaces. It was summarized that the influence of particle agglomeration degree on the properties of composites with various particle volume fraction.

2. Experiment and modeling

2.1. Materials and experiment
The commercial AA6016Al alloy was selected as the matrix material. For the synthesis of the ZrB2/AA6016Al alloy composites, inorganic salt K2ZrF6 and KBF4 powders were chosen as reaction sources for formation of ZrB2 by DMR method. The typical procedures have been described in the previous works [19]. The mixed inorganic salt powders were added into the aluminum alloy melt at 850 °C and isothermally held for half hour, during which an electromagnetic stirrer was utilized to increase mass transfer. 1 vol%, 2 vol% and 3 vol% ZrB2/AA6016Al composites were prepared and the microstructure is exhibited in figure 1.

2.2. Modeling
The FEA is based on the RVE, which presumes the microstructure of composites can be reconstructed by assembling such elements. In order to analyze micro- and macro-mechanical response of in situ nanoparticles reinforced composites, it is primary to investigate sizes, morphology and distribution of particles and aggregated particles degree of clusters. Above information are observed from SEM analysis of 1 vol%, 2 vol% and 3 vol% ZrB2/AA6016Al composites, as shown in figure 1. Figure 2 presents that a distinct interface layer wasn’t be observed under TEM, therefore the interface layer with thickness is not created in this study. The particles are connected with the matrix in a fully integrated way.

Due to a clustered region as an integral group, it can be supposed that clusters occur the elastic-plastic deformation instead of the elastic deformation. In-situ ZrB2 particles are considered as elastic-brittle phases and elastic-plastic behavior is within the 6016 Al alloy matrix. Consequently, both the clustered regions and the matrix are assumed to elastic-plastic. For the strain hardening of material, the property is expressed by a nonlinear fitting function of the Voce type written as equation (1) [20],

$$\sigma_{eq} = \sigma_0 + R_0(\varepsilon - \varepsilon_0) + (\sigma_{max} - \sigma_0)[1 - e^{-b(\varepsilon - \varepsilon_0)}]$$

where $\sigma_{eq}$ is the equivalent plastic stress, $\sigma_0$ and $\sigma_{max}$ are yield limit and tensile limit, respectively, $\varepsilon_0$ represents elastic limit strain, $R_0$ and $b$ are fitted through the stress-strain curves.

To model the fracture behavior of composites, a progressive Hashin damage criterion [21] is used to predict the onset of damage. As the second-phase particles, ZrB2 nanoparticles do not fail in the way of ductile fracture or brittle fracture [22], so the failure mechanism of ZrB2 is not considered. It is assumed that the damage initiates until the value of the following quadratic interaction function reaches a value of one, as
For the special case of in-plane models under the uniaxial loading, $\eta_{13}$ is neglected in this research. Moreover, a damage evolution law [23], written as equation (3), is applied to describe the degradation of the material stiffness matrix after damage initiation:

$$
\left( \frac{\eta_{13}}{X_T} \right)^2 + \left( \frac{\eta_{12}}{S_{12}} \right)^2 + \left( \frac{\eta_{33}}{\gamma_{13}} \right)^2 \geq 1
$$

(2)

Figure 1. SEM micrographs of in situ ZrB$_2$/AA6016 composites: (a) 1 vol%; (b) 2 vol%; (c) 3 vol% and (d) 3 vol% in situ ZrB$_2$/AA6016 composite at higher magnification.

Figure 2. TEM micrographs of in situ ZrB$_2$/AA6016 composites: (a) the particle agglomeration; (b) the particle-matrix interface.
\[ L_{22} = 1 - \frac{\bar{\sigma}_{22}}{\bar{C}_{22} \bar{\epsilon}_{11} + \bar{C}_{22} \bar{\epsilon}_{22}} \]

\[ L_{66} = 1 - \frac{\bar{\sigma}_{12}}{\bar{C}_{66} \bar{\epsilon}_{12}} \]

where \([\bar{\mathbf{C}}]\) is the initial in-plane stiffness matrix and \([\mathbf{C}]\) is the reduced in-plane stiffness matrix. In the process of uniaxial tensile simulation, the displacement loading is applied continuously until the calculation results are not convergent.

The performance of clustered regions with different local aggregated degree is going to be the first object to be simulated in which the diameter of ZrB\(_2\) particles is fixed at 80 ± 10 nm which nearly covers the diameter range of ZrB\(_2\) particles. In the current work, nano particles are described by irregular convex polygons and cannot overlap, which is realized by controlling distances between particles. The Monte Carlo algorithm is employed to randomly deposit particles without overlapping with each other in the aluminum alloy matrix. One convex polygon is defined by the reference points \((X_0, Y_0)\), edge numbers \(N\), polar angle \(\alpha\) and polar Radius \(R\).

Edge numbers of convex polygons are controlled by equation (6), which is given in the form

\[ N = N_{\text{min}} + \text{round}[\eta(N_{\text{max}} - N_{\text{min}})] \]

where \(\eta\) is a random number generated by computer with uniform distribution between 0 and 1, \(N_{\text{max}}\) and \(N_{\text{min}}\) are the maximum and minimum of polygon edges, \(\text{round}\) is an integral function. As figure 1(d) showed, the edge number is almost between 4 and 6. Therefore, in this work, \(N_{\text{max}}\) and \(N_{\text{min}}\) take the value of 6 and 4, respectively.

Polar Radius \(R\) is a random number between 35 nm and 45 nm. Polar angle \(\alpha\) is determined by the number of edges, i.e.,

\[ \alpha = \frac{2\pi}{N} \]

The local particulate aggregated degree of one representative cluster element can be drawn by the particle volume fraction of one individual clustered region. Here the degree \(\zeta\) of aggregated particles describe as follows,

\[ \zeta = \frac{V_C^p}{V_C} \]

where \(V_C^p\) is the volume of particles in one cluster and \(V_C\) is the volume of one cluster. Figure 3 depicts that the clusters possess different local particle volume fraction. Therefore in this research three types of cluster model with different agglomerated ratios are constructed. The corresponding representative cluster element models (1 \(\mu\)m × 1 \(\mu\)m) are shown in figure 4 where agglomerated ratios were 15%, 20% and 25% respectively.

Considering various agglomerated ratios, composites with different particle volume fractions are simplified into nine groups of RVE models (100 \(\mu\)m × 100 \(\mu\)m). The number of clusters in the composite RVE models is controlled by the combination of \(\zeta\) and particle volume fraction \(f\), as shown by equation (9),

\[ n = \frac{100^2 \times f}{\pi \times r^2 \times \zeta} \]

where \(r\) is the diameter of the cluster. The coordinates of cluster centers are also determined by the Monte Carlo algorithm. Figures 5 and 6 are the number of clusters of FEMs and the corresponding RVE models respectively. The displacement loading is applied to all RVE models in the X axis direction and all three rotation degrees of all nodes are constrained. Material parameters of the matrix and ZrB\(_2\) particles [24] are given in table 1.
3. Results and discussion

3.1. Effects of particles on elastic-plastic deformation and damage behavior

The simulated elastic-plastic behavior of representative cluster element models with different agglomerated ratios is shown in figure 7. The ultimate strain decreases apparently with increasing aggregated ratio of particles, while the elastic modulus and yield strength are not significantly affected by increasing agglomerated ratio besides slight increase. Parameter $R_0$ and $b$ of three models were fitted by Voce type nonlinear hardening model, and the specific values are shown in table 2. The numerical results of curve fitting indicate that the cluster model with higher particulate aggregated ratio shows a higher yield strength $\sigma_0$ and a lower elastic limit strain $\epsilon_0$ than the cluster with lower aggregated ratio of particles. Under the same condition of strain, the corresponding stress raises with the increase of agglomerated ratio of particles, yet the maximum strength is similar.

As depicted in figure 8, the damage behavior of RVE models is discussed in this study. From the figures 8(a)–(c), it can be concluded that damage initiates in those matrix elements which are adjacent to the particles. The damage initiation points mostly occurred at the sharp corners of the particles pointing in the identical direction as the uniaxial tensile. In the meantime a series of little local damages caused by stress concentration happened in the matrix, figures 8(d)–(f), finally contributing damages to propagate into the whole matrix. Once one or more cracks cross through the entire matrix, dividing it two or several parts, materials fail to resist deformation and eventually break, as shown in figures 8(g)–(i). Figure 9 presents the Von Mises stress of representative cluster elements with different degree of particle segregation when stress reached the maximum strength. And it is also found in figure 9 that there is not large stress concentration and obvious strain inside the particles, so it can be demonstrated that the nano-scale particles will not fail in the way of particle cracking which micro-scale particles happen. Above results indicate that nano particles play an important role in blocking dislocation movement and deformation of composites.

![Figure 4. Representative cluster element models with various agglomerated ratios: (a) 15%; (b) 20%; (c) 25%.

![Figure 5. The number of clusters of composite RVE models.](image)
The second-phase particle strengthening is a primary strengthening mechanism that could contribute to considerable strength increases in the PRMMCs. For Orowan strengthening, there is an inverse relationship between the strength enhancement and the interparticle spacing. The distance between adjacent particle centers was investigated in the three models of figure 3, as shown in table 3. The increase of agglomeration degree in unit volume will directly induce the decrease of particle spacing. Therefore, yield strength $\sigma_0$ is elevated with increasing agglomerated ratio.

Table 1. Material parameters of the composite constituents.

|                      | AA6016 (As-cast) | ZrB$_2$ |
|----------------------|------------------|---------|
| Young’s modulus $E$ [GPa] | 68               | 495     |
| Poisson ratio $\nu$    | 0.3              | 0.13    |
| Yield strength $\sigma_0$ [MPa] | 119               |         |
| Ultimate strength $\sigma_{\text{max}}$ [MPa] | 180               |         |

Figure 6. RVE models of composites with different agglomerated ratios: (a), (b) and (c) 1 vol% ZrB$_2$/AA6016 composites; (d), (e) and (f) 2 vol% ZrB$_2$/AA6016 composites; (g), (h) and (i) 3 vol% ZrB$_2$/AA6016 composites.
3.2. Effects of clusters on deformation and mechanical properties of composites

The mechanical properties of 1 vol%, 2 vol% and 3 vol% nanoparticles reinforced aluminum matrix composites with various agglomerated ratios were simulated. It is worth reiterating that here we regard one cluster as an integral which possesses elastic-plastic properties. Nine groups of stress-strain curves are shown in figures 10(a)–(c). In the elastic deformation stage, the experimental values of elastic modulus are in good agreement with simulation results. For PRMMCs, the Halpin-Tsai model possesses impressive accuracy in the prediction of elastic modulus and takes the following form:

\[ E = \frac{E_m(1 + 2qf_p)}{1 - qf_p} \]  
\[ q = \frac{E_p - E_m}{E_p + 2E_m} \]

Equation (10) indicates that the elastic modulus of composites is not related to the distribution of particles. In model 1 ~ 9, the following relationship exists between agglomerated ratios \( \zeta \) and particle volume fraction \( f_p \):

\[ \sum_{i=1}^{n} \zeta_i V_i = f_p \]  

It keeps the final Halpin-Tsai equation of this research model same as equation (10). Therefore, although various agglomerated ratios are considered into the composites models, the elastic modulus of composites do not change.

The enhancement of yield strength is mainly attributed to the load bearing strengthening, Orowan strengthening, modulus mismatch strengthening, coefficient of thermal expansion (CTE) mismatch strengthening [26]. The thermal expansion mismatch between the matrix and clusters is neglected in this study. The strengthening effect of \( \Delta \sigma_{load} \) can be calculated by equation (13)

\[ \Delta \sigma_{load} = 0.5V_p \sigma_m \]

where \( V_p \) is the particle volume fraction and \( \sigma_m \) is the yield strength of the matrix. Orowan strengthening is normally expressed by the following relationship as,
Figure 8. The damage behavior in the RVE models: (a), (d) and (g) the 15% agglomerated ratio model; (b), (e) and (h) the 20% agglomerated ratio model; (c), (f) and (i) the 25% agglomerated ratio model.

Figure 9. (a) Von Mises Stress distribution of 15% agglomerated ratio model; (b) Von Mises Stress distribution of 20% agglomerated ratio model; (c) Von Mises Stress distribution of 25% agglomerated ratio model.

|        | 15%             | 20%             | 25%             |
|--------|-----------------|-----------------|-----------------|
| Range  | 0.113 ~ 0.229μm | 0.1018 ~ 0.209μm| 0.091 ~ 0.1947μm|
\[ \Delta \sigma_{\text{Orowan}} = \frac{AG_mb_m}{2\pi\lambda} \ln \left( \frac{d_p}{r} \right) \]  
(14)

where \( A \) is constant depending on the materials, \( G_m \) is the shear modulus, \( b_m \) is the Burgers vector, \( \lambda \) is the interparticle spacing, \( d_p \) is average particle diameter and \( r \) is the dislocation core radius.

The strength enhancement of the modulus mismatch strengthening can be estimated by equation (15)

\[ \Delta \sigma_{\text{modulus}} = \alpha G_m b_m \sqrt{\rho_{\text{modulus}}} \]  
(15)

\[ \rho_{\text{modulus}} = \frac{8G_m}{b_m\lambda} \]  
(16)

where \( \alpha \) is a material-specific coefficient and \( \rho_{\text{modulus}} \) represents the density of geometrically necessary dislocations (GND). However, it must be noticed that Orowan strengthening is effective only when sizes of particles are lower than 1 \( \mu m \) [27]. Thus, Orowan strengthening cannot be applied to composites models reinforced by simplified particulate clusters in this research. The total yield strength improvement is estimated by the quadratic summation method [28], which is given in the form:

\[ \Delta \sigma = \sqrt{\Delta \sigma_{\text{modulus}}^2 + \Delta \sigma_{\text{Load}}^2} \]  
(17)

The simulation results of model 1 ~ 9 indicate that finite element analysis is performed to predict the Young’s modulus of composites, which is in good agreement with the experimental results. Yield strength among 9 groups of models is a similar value, but lower than the experimental results, which illustrates that the mechanism of load transfer strengthening merely considered in this research is not enough to fully reflect the actual strengthening effect.

For composites with the same agglomeration degree, the higher particle volume fraction attributed to the lower elongation. This tendency is in good agreement with actual experimental results. The elongation of composite models with the same particle volume fraction decreases monotonously with increasing agglomerated ratios. These results also reveal that it is feasible that the particulate cluster was considered as an integral reinforcement. The agglomeration degree of particles directly affects the size of the interparticle spacing and results in the different mechanical properties of the PRMMCs.

Figure 11 depicts the evolution of Von Mises stress distribution of the model 9 under the uniaxial tensile loading. It can be seen that clusters bore more loading at the initial stage of deformation and gradually generated stress concentration. After stress arrived at ultimate strength, as presented in figures 9(c) and (d), multiple high stress concentration regions gradually expanded and contacted each other to form a large stress concentration.
area. In figure 9(d), the stress concentration penetrated the cluster and matrix and achieved the maximum inside the cluster. The evolution of stress demonstrates the reason why the fracture morphology of composites is mostly quasi-cleavage fracture.

Consequently, ductile fracture characteristics of metal matrix are generally detectable in actual fracture surface. The SEM micrographs in figure 12 reveals the morphology of fracture section, where a large number of tear ridges can be observed. Moreover, fracture surfaces of ZrB$_2$/AA6016 composites revealed that there are ZrB$_2$ particles in the dimples (figure 13). This phenomenon is caused by dislocation pile-up that exists outside the agglomeration area of nanoparticles. Therefore, these observations provide support to the validity of the models selected for reconstructing the in situ ZrB$_2$/AA6016 composites, and suggest that the proposed numerical model can be applied to predict stress-strain response of composites.

Compared with conventional particle reinforcements, the in situ nanoparticle reinforcements considerably influence the properties of composites from the following aspects. First of all, it is commonly accepted that the in situ PRMMCs possess strong interfacial bonding. Because of the better conjunction between the nanoparticles and the matrix, the reinforcements can delay the debonding of particles and bear more loads. Furthermore, the reduction of the grain size by existence of nanoparticles resists the dislocations movement across grain.

Figure 11. Von Mises stress distribution of model 9 during tensile deformation.

Figure 12. Fracture surfaces of (a) 2 vol% in situ ZrB$_2$/AA6016 composites and (b) 3vol% in situ ZrB$_2$/AA6016 composites.
boundaries. On the other hands, nanoparticles themselves can work as impediments during the motion of dislocations. All of the above advantages improve the strength of the composites in varying degrees. However, the inclination of particles aggregation increases obviously with particle volume fraction increasing, which lessens the elongation. The outcomes of figures 8 and 11 directly present the positive influence and repercussion of nanoparticles reinforcements on the properties of composites.

4. Conclusions

In this work, nano-ZrB$_2$/AA6016 Al alloy composites as a model material, the effect of particles on deformation behavior and the effect of agglomerated degree on mechanical properties of composites were investigated by combining finite element simulation with experiments. The cluster was supposed as an entirety in composites, which reduces the complexity of finite element modeling of composites, thus enlarging the size of finite element model and lowering the meshing distortion caused by the tiny size of nanoparticles during meshing. The mechanical response of the cluster was simulated by FEA under the uniaxial tension condition. The main conclusions can be summarized as

(1) The increase of agglomerated ratio hastens the stress concentration. The damage originates from the interface between particles and the matrix and occurs mostly at the sharp corners of the particles pointing in the identical direction as the tensile. Not failing in the way of particle cracking, so nanoparticles play a crucial role in blocking dislocation movement and deformation.

(2) The load transfer enhancement caused by the stiffness mismatch strength and load bearing strength is limited and is not enough to fully reflect the actual strengthening effect.

(3) The predicted value of Young’s modulus of composites is consistent with the experimental results. For composites with the same agglomeration degree, the higher particle volume fraction attributed to the lower elongation. With increasing agglomerated ratios, the elongation of composite models with the same particle volume fraction decreases monotonously.

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