Int J Adv Manuf Technol (2015) 79:1905–1914
DOI 10.1007/s00170-015-6928-2

ORIGINAL ARTICLE

Grain size effect of thickness/average grain size on mechanical behaviour, fracture mechanism and constitutive model for phosphor bronze foil

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Received: 1 September 2014 / Accepted: 20 February 2015 / Published online: 12 March 2015

Abstract Size effects play a significant role in microforming process, and any dimensional change can have a great impact on materials' mechanical properties. In this paper, the size effects on deformation behaviour and fracture of phosphor foil were investigated in the form of grain size effect: the ratio of materials' thickness (*T*) to average grain size (*D*) by micro tensile tests. The ratio was designed to be closed to but larger than, less than and equal to 1, respectively. The results show that the amount of plastic deformation decreases with the decrease of the ratio of *T*/*D*, which indicates that the grain size plays a significant role and grain deformation modes differ when the ratio changes. It is also found that their fractograph reflects different features in terms of micro-dimples and cleavage planes, further demonstrating that when *T*/*D* >1, its materials have a tendency to fracture ductilely, while materials would like to conduct brittle fracture when *T*/*D* <1. So the ratio of *T*/*D* which is close to 1 can be regarded as the divide of ductile fracture and brittle fracture. For *T*/*D* <1, a new constitutive model is proposed based on the classic composite model. The model's results are compared with the experimental ones and the efficiency of the developed models is verified.

Keywords Size effect · Thickness/average grain size · Mechanical behaviour · Fractograph · Constitutive model

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1 Introduction

There is no doubt that microforming technology has attracted tens of thousands of attentions due to the prevalent usage of its matching products-micro parts. The increasing demand of micro-scale products for high accuracy and high quality has also inspired the development of microforming technology. Although the metal forming in traditional level has been progressed for centuries and a series of classic knowledge and theories have been established systematically by previous researchers, it is commonly known that these macro-scale processing theories cannot be directly applied in micro-scale world with miniaturisation due to size effect [1–3]. When specimens are scaled downsize to micro-level, the parameters which may not be important in conventional process begin to play a significant role in controlling the accuracy of deformation, and in another way, determining the dimensions of processed specimens.

Several researches have been conducted to investigate the size effect in micro deformation behaviours and mechanics. Engle and Eckstein [1] proposed the surface layer model in which the specimen was divided into two portions, interior part and surface layer, to explain the reduction of flow stress in microforming process. Geiger et al. [4] and Vollertsen et al.

[5, 2] published papers with respect to microforming and carried out a comprehensive review of state of the art of microforming technology. Fu et al. updated the development of these microforming technologies in the latest review [6]. Diehl et al. [7] systematically investigated the relationship between the microstructure and mechanical properties and the forming behaviour by tensile and hydraulic bulge tests and bending test on thin metal foils. Ma et al. [8] researched the size effect on fracture behaviour in deep drawing process. Their results illustrate that limit drawing ratio (LDR) is significantly decreased with grain size. Kals and Eckstein [9] investigated the size effect in tensile tests, air bending and punching of sheet metals by miniaturisation based on similarity theory, and they concluded that the deformation behaviour in micropunching is strongly different from those in both tensile test and air bending. Further research from Raulea et al. [10] showed that the yield strength is related to the ratio between the grain size and specimen thickness, which was also demonstrated in a planar blanking and bending process. Chan et al. [11] investigated the scatter effect of grain mechanical properties with micro-compression process and proposed a finite element model with the consideration of grain size and the scatter effect of flow stress, which provided a basis for understanding and modelling of material size effect in microforming process. Moreover, they investigated the interactive effect of specimen and grain sizes on deformation behaviour [12] and the size effect of pure copper in micro extrusion process [13]. Lu et al. [14] enhanced this modelling by implanting Voronoi tessellation algorithm into preprocessor of finite element software. Moreover, Xu et al. [15, 16] studied the size effect on deformation behaviour and fracture feature by micro-blanking in terms of the ratios of different blanking clearances to grain size, $C/D > 1$, $C/D \approx 1$ and $C/D < 1$, and found out that the ultimate shearing strength reaches an extreme value when blanking clearance to grain size ratio is equal to 1. Wang et al. [17] investigated the size effect of cavity dimension in coining process. They revealed that the micro-formability is decreased with the increase of the ratio between grain sizes and die cavity width. Gau et al. [18] conducted three-point bending to study the correlation between springback amount and thickness/average grain diameter (T/D) ratios. They observed that springback is related to the ratio of sheet thickness to grain size, but all the ratios were larger than 1. Liu et al. [19] developed a constitutive model considering the grain boundary strain hardening. Yeh et al. [20] also proposed a mathematical model which took into account the thickness and grain size effects in micro cup drawing. Fu et al. [21] studied the size effect on fracture behaviour of the annealed copper foils with different thicknesses and grain sizes by tensile tests, and they found that the number of micro-voids as well as the fracture stress and strain decreases with T/D. Hmida et al. [22] conducted the single point incremental forming process of copper foils with different grain sizes, and it is observed that the formability decreases with T/D. Chan and Fu [23] carried out experimental studies and numerical modelling on the flow stress of sheet metal in microforming in terms of T/D. Michel and Picart [24] also conducted tensile and hydraulic bulging tests on CuZn36 to evaluate size effects on the constitutive behaviour. However, the ratios of thickness to grain size in these studies are limited in greater than 1. Yun et al. [25] proposed a constitutive model for thin metal considering the first order size effects, but they found that the great offset occurred when $T/D < 1$. Peng et al. [26] adopted a uniform double linear constitutive equation into their model to study the constitutive behaviour in microforming and proposed a uniform size-dependent constitutive model by introducing a scale factor to evaluate the material behaviours [27].

In microforming process, the size effect of materials plays a significant role in influencing the deformation results and the accuracy of final products. Nevertheless, it is noticeable that among the prior studies, there is a lack of in-depth research on the interactive effect of micro deformation and fracture behaviours, and the size effect expressed by the ratios of specimen thickness to grain size, especially when they are approximately larger than, equal to and less than 1. This means that there is just more than one grain, only one grain or an incomplete grain in the thickness direction. In this study, uniaxial tensile tests were carried out with phosphor bronze foil to study the effect of the ratio T/D on micro-scale plastic deformation, and their fracture morphologies were observed under scanning electron microscope (SEM). The ratios are reflected as $T/D > 1$ (1.23), $T/D < 1$ (0.68) and $T/D \approx 1$ (1.07), respectively, by means of different annealing treatments. At the same time, with consideration that the surface layer model cannot be applicable when $T/D < 1$, a new constitutive model are proposed based on the classic composite model. The results obtained from the model are compared with the experimental ones, and the efficiency of the developed model is verified.

2 Experiment

2.1 Sample preparation

Phosphor bronze C5191 foil from cold rolling with thickness 70 μm was employed to conduct this study due to its wide application in micro fabrication. The chemical compositions of phosphor bronze are displayed in Table 1.

Table 1 Chemical compositions of phosphor bronze C5191, in wt%

Sn	P	Pb	Fe	Zn
5.87	0.22	0.004	0.001	0.004

The grain size effect which is the interactive effect between the specimen and grain size is expressed by the ratio of the specimen thickness to average grain size:

$$\text{Grain size effect} = \frac{\text{specimen thickness } (T)}{\text{average grain size } (D)} \quad (1)$$

To realise the grain size effect which is greater than, less than and equal to 1 individually, the materials were annealed with different temperatures and holding times in Ar air protection condition before furnace cooling. The anneal conditions are presented in Table 2. The heat-treated specimens were etched using a solution of 5 g of $FeCl_3$, 50 ml of HCl and 100 ml of H_2O for 1–2 s. The microstructures of three different grain sizes in thickness direction are shown in Fig. 1. It can be seen that the grain size increases with holding time under the same annealing temperature. The average grain size was obtained by the software ImageJ. Straight line was drawn along a longitudinal direction in a microstructure image. The line was both starting from and ending at the boundary of a certain grain. The length of the line was L, and the quantity of the grains which were intercepted by the line was N ($N \geq 30$). Then the average grain size was obtained as L/N. Total ten samples were observed and measured in this way for each group. The largest and smallest values were discarded, and the final average grain size was the average value for the rest of the eight samples.

After confirming annealing schedules, the original specimens were sectioned to dog-bone shape which can be seen in Fig. 2 by a wire-cut electrical discharging machine (EDM). Then the cut specimens were heat treated to be featured with the different grain size effect ratios: T/D <1, $T/D \approx 1$ and T/D <1. This operation (annealing after sectioning) can avoid the machining stress during EDM since the material is so thin.

2.2 Micro tensile test

To further investigate the grain size effect on the mechanical properties and fracture mechanism, micro tensile tests were conducted and the specimens were elongated till fracture. The tests were conducted on Instron micro tester 5848 as shown in Fig. 3. This equipment can offer ultra-precise displacement control, 20 nm, and can accommodate ultra-thin

Table 2 Heat treatment and average grain size

Temperature (°C)	550	550	550
Time (h)	4	6	8
Thickness, T (μm)	70	70	70
Average grain size, D (μm)	57.1	65.7	103.4
T/D	1.23	1.07	0.68

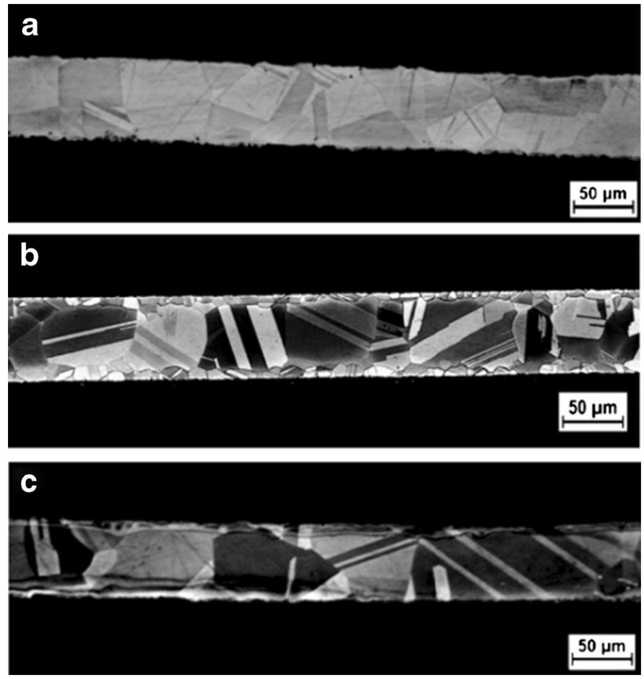

Fig. 1 Microstructure of annealed phosphor bronze foil. **a** T=70 μm, D=57.1 μm, T/D <1; **b** T=70 μm, D=65.7 μm, $T/D \approx 1$; **c** T=70 μm, D=103.1 μm, T/D <1

components. A video extensometer is also installed to ensure the accuracy of stress/strain measurements. The crosshead velocity was 0.02 mm/s applied for all the tests, and each test was repeated three times.

3 Results and discussion

3.1 The effect of T/D on stress-strain curves

It can be seen from Fig. 4 that material's plasticity increases with the increase of their ratios of T/D, while the scatter of stress-strain curve profile decreases. When T/D <1, the average grain size is larger than specimen's thickness, which means that most grains involved in the tensile specimens are incomplete. Consequently, due to the lack of grain boundaries

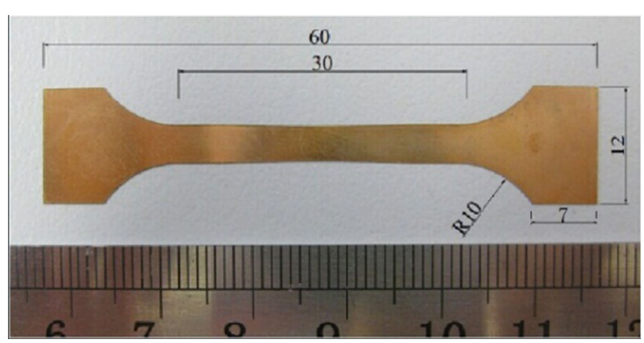

Fig. 2 The tensile test specimen

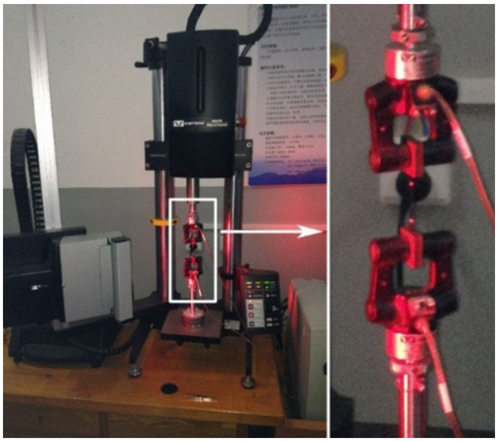

Fig. 3 Instron micro tester 5848 with video extensometer

and grain boundary corners, concentrated plastic deformation is very difficult to be initiated [28]. This can explain that tensile tests in this group hardly experience plastic deformation. Furthermore, it is clear that this ratio's scatter of stress-strain curves is most obvious in these three groups shown in Fig. 4a. This is because without the limitation of grain boundaries, the grain crystal orientation varies and the sliding systems are oriented distinctively when starting an applied stress. In addition, some other deformation modes, such as grain rotation and coordination, could change the grain deformation behaviours. Therefore, this kind of grain heterogeneity can contribute to the irreproducible strain-stress relationships. When *T*/*D* >1, the specimen materials can be regarded as polycrystalline aggregate. The quantity of grain boundaries and grain boundary corners will increase largely, which will lead to the appearance of work-hardened grain boundary layers [29]. Once these layers are formed, the stresses within the polycrystalline aggregate will homogenise. The deformation normally takes place with the most favourable grain sliding systems; thus, the strain-stress curves obtained from this group are the most reproducible which is reflected in Fig. 4c. At the same time, due to the large number of dislocations piled up at grain boundaries and their corners, there is a great tendency to activate the plastic deformation, so materials with the ratio *T*/*D* >1 have the best plasticity compared to the other two. When *T*/*D*≈1, it is notable in Fig. 4b that stress-strain

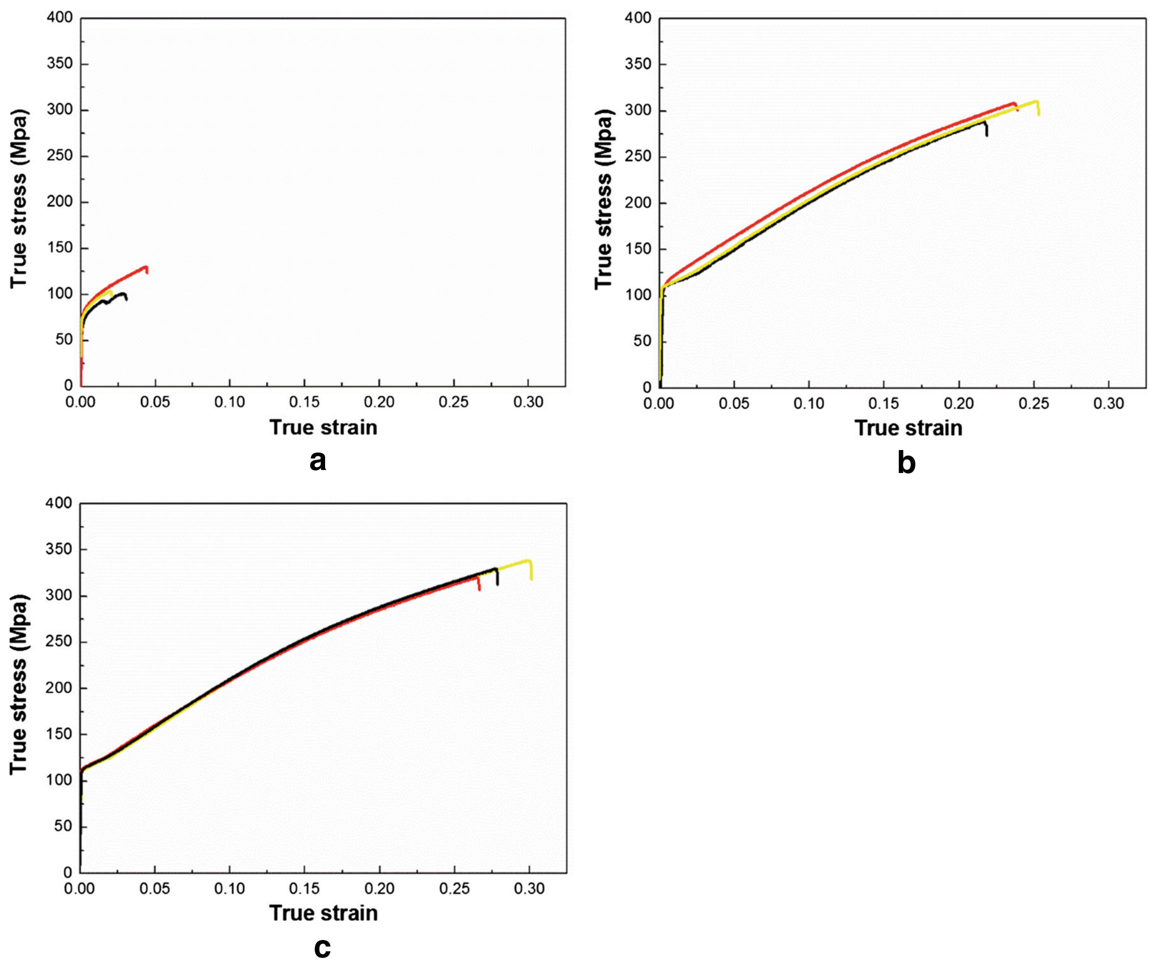

Fig. 4 Strain-stress curves with different *T*/*D* ratios: **a** *T*/*D* <1, **b** *T*/*D*≈1, **c** *T*/*D* >1

curves are repeated roughly. In this case, there is only one grain in the thickness direction; thus, grain matrix deformation and grain boundary sliding can be dominant simultaneously during tests [15]. Plastic deformation is relatively easy to happen because there are grain boundary corners gathering near two sides of thickness direction. However, due to the limitation from thickness direction, grain orientation and coordination cannot occur as easily as they are compared to materials with *T*/*D* >1. Therefore, this uncertainty can attribute to the slight scatter of stress-strain curves. According to the results from tensile tests, it is found when the ratio of thickness to grain size is small enough (around 1), and the strain-stress relationship on the one hand is consistent with classic Hall-Petch relationship as long as average grain size is not scaling down to 10 nm [30]. On the other hand, it becomes very sensitive to the value of the ratio of thickness to average grain size, especially when comparing the plastic deformation occurred with material's *T*/*D* >1 to *T*/*D* <1.

3.2 The effect of *T*/*D* on fracture behaviour

The fractograph of the tensile-tested samples are shown in Fig. 5. It is noticeable that different fractograph images were obtained with different ratios of thickness to average grain size. It is found that the number of micro-dimples decreases with the decreasing *T*/*D*. This is consistent with prior studies [21]. The grain boundary fraction in the material increase with the value of *T*/*D*, and because the grain boundaries act as obstacles to dislocation motion, the stresses concentrate at or near grain boundary regions and cause micro-void and micro-dimple formation consequently in the deformation process. At the same time, micro-dimples and cleavage planes can be found together in all three fractograph, which shows materials can have the mixture of ductile fracture mode and brittle fracture mode, indicating that partial plastic deformation occurred during tensile testing. However, a difference can be observed on the fracture surface that the micro-dimples generated when *T*/*D* >1 is averagely distributed along fracture direction, while when *T*/*D*≈1, micro-dimples are mainly focused on a certain limited area, and only a few micro-dimples can be found when *T*/*D* <1. This phenomenon shows that when more than one grain exists in thickness direction, its material's plasticity is better than that of the material which has just one grain or incomplete one in thickness direction. Inversely, when the ratios of *T*/*D* decrease from larger than 1 to less than 1, the number of the cleavage planes increases gradually. When *T*/*D* <1, cleavage planes take up almost the whole area of fracture surface. This dramatic change can be explained when the

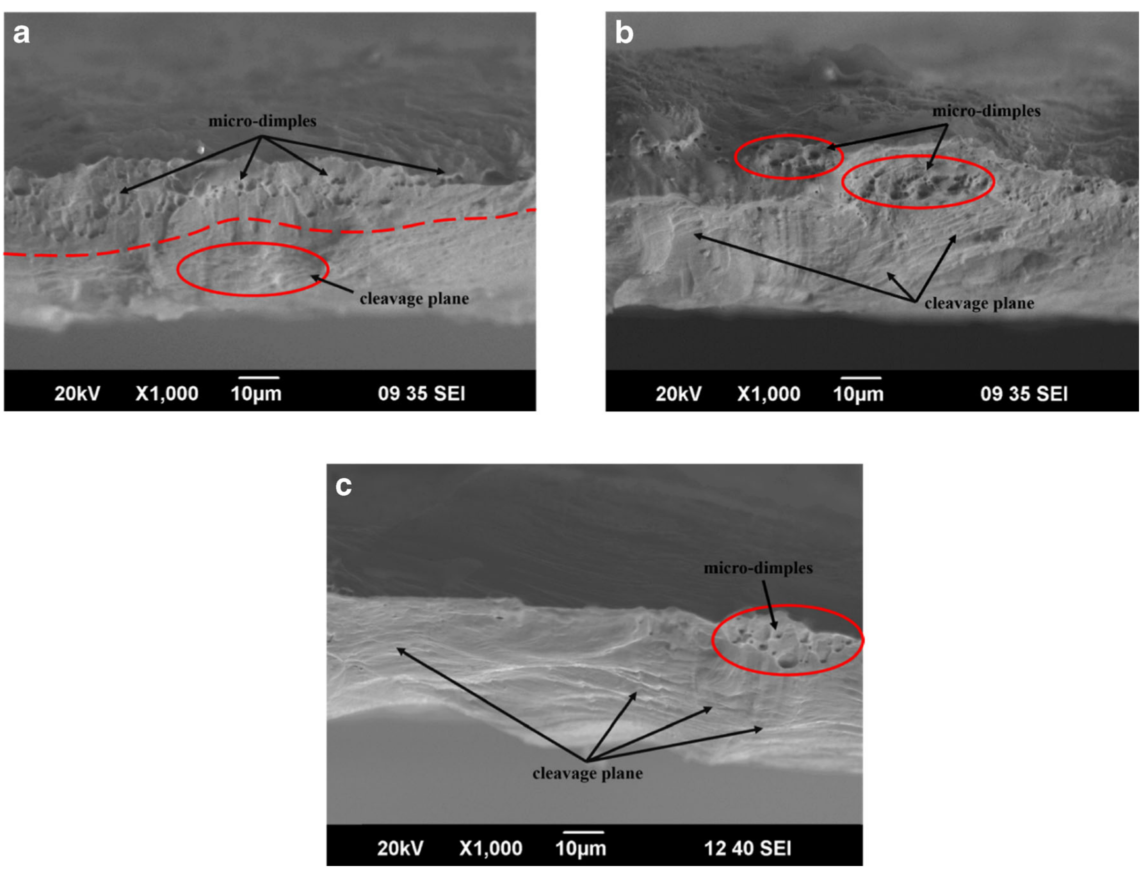

Fig. 5 Fracture morphology of tested samples. **a** *T*/*D* >1, **b** *T*/*D*≈1, **c** *T*/*D* <1

grain size approaches to, even overpass the thickness value, the location, and the size and orientation of each grain affect the fracture behaviour significantly. With the ratio T/D decreases, the quantity of the grain boundaries will also decrease, which means that when only incomplete grains exist in thickness direction, it forces the grains to have an extremely unfavourable orientation towards the tensile direction, leading to a strong tendency of brittle fracture.

4 Constitutive model

Normally, the surface layer model is applied when there are several grains in the thickness direction, which means that T/D is comparatively greater than 1. From Fig. 6, it is illustrated that when T/D >1, the specimen can be divided into two portions: volume (inner) grains and surface grains.

However, in our study, the thickness value is close to the average grain size. When the ratio of T/D is slightly greater than 1, there is generally at least one complete grain and a partial grain in thickness direction. So the material still can be regarded as the above two portions. The one grain can be taken as the inner grain of the material, and the other portion can be treated as surface grain. Therefore, the classic surface layer model can still be applicable in this way. The flow stress of the material is expressed as the weighted average of the inner portion' stresses and the surface layer portion's stresses as follows:

$$\sigma = \eta\sigma_{\mathrm{inner}} + (1-\eta)\sigma_{\mathrm{surf}} \tag{2}$$

where σ is the flow stress of the material and σ_{inner} and σ_{surf} are the stresses of the inner portion and the surface layer of the material, respectively. η is the size factor to express the fraction of inner portion to the whole material area. It can be calculated based on the following equation [31]:

$$\eta = \frac{T-D}{T} \tag{3}$$

where T is the thickness of the specimen and D is the average grain size.

It is notable that this equation cannot be applied when $T<D$ (T/D <1), which means the grain size is greater than the thickness, and only incomplete grains exist in the cross section of material. When T/D >1, the assumption that the flow stress of surface layer is equal to that of grain interior was adopted in the description of surface layer model [19, 14]. However, this is not applicable because the grain boundary strengthening effect cannot be neglected due to the limited quantities of grain boundaries in this circumstance. Since there is no good way to divide the T/D <1 microstructure into two portions just like the classic surface layer model, a new constitutive model is proposed in order to better describe this particular grain size effect.

The methodology of studying this extreme grain size effect on flow stress is based on the composite model developed by Kocks [32] and Meyers and Ashworth [29]. This model has been proven to be a valid and effective approach to describe the relationship between the flow stress of polycrystalline aggregate and grain size. This composite model assumes that grains are spherical and are composed of grain boundary layers and grain interiors, which is illustrated in Fig. 7. The idealised representation of the aggregate for T/D <1 is shown in Fig. 8, and the flow stress of the grain aggregate can be expressed as:

$$\sigma_y = A_{\mathrm{G}}\sigma_{f\mathrm{G}} + A_{\mathrm{GB}}\sigma_{f\mathrm{GB}} \tag{4}$$

where A_{G} and A_{GB} are the areal fractions of grain interior and grain boundary, respectively. With average grain size D and material thickness T, the grain boundary layers are assumed to have a thickness t.

As it can be seen from Fig. 7 that an idealised spherical grain is limited in thickness direction, and the proportion of grain interior and grain boundary is determined by the thickness value of the material and average grain size. These parameters can be quantified as the

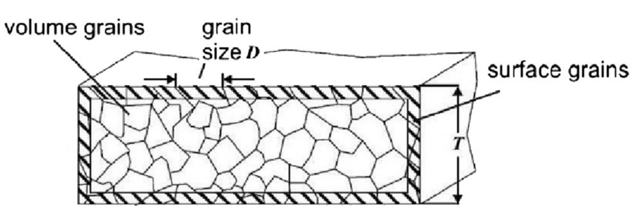

Fig. 6 The share of the volume grains and surface grains

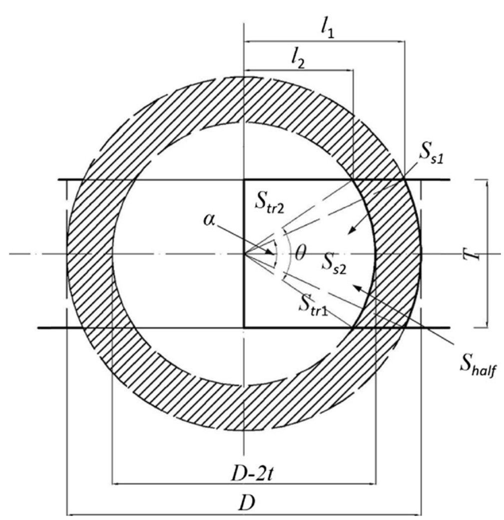

Fig. 7 Schematic of proportions of grain interior and grain boundary

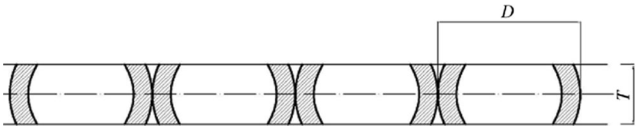

Fig. 8 Ideal aggregate viewed as composite materials with single incomplete grain in thickness direction (T/D <1)

ratio of T/D. Taking the right half grain as an example, the areal fractions are expressed by:

$$A_G = \frac{S_1 - S_2}{S_{half}} \tag{5}$$

$$A_{GB} = \frac{S_{half} - S_1 + S_2}{S_{half}} = 1 - A_G \tag{6}$$

where S_{half} denotes the area of right half rectangle, and area S_1 consists of one sector S_{s1} and two triangles S_{tr1}. Similarly, area S_2 is made up of one sector S_{s2} and two triangles S_{tr2}, and the extra part of the rectangle is treated as grain interior.

$$S_{half} = T \cdot D/2 \tag{7}$$

$$S_1 = S_{s1} + 2S_{tr1} \tag{8}$$

$$S_2 = S_{s2} + 2S_{tr2} \tag{9}$$

The two sectors S_{s1} and S_{s2} can be calculated respectively as:

$$S_{s1} = \frac{\theta \pi \left(\frac{D}{2}\right)^2}{360} \tag{10}$$

$$S_{s2} = \frac{\alpha \pi \left(\frac{D}{2} - t\right)^2}{360} \tag{11}$$

where θ is the central angle for S_{s1} and α is the central angle for S_{s2}. They are expressed based on a law of cosines by:

$$\theta = \cos^{-1}\left[\frac{2\left(\frac{D}{2}\right)^2 - T^2}{2\left(\frac{D}{2}\right)^2}\right] \tag{12}$$

$$\alpha = \cos^{-1}\left[\frac{2\left(\frac{D}{2} - t\right)^2 - T^2}{2\left(\frac{D}{2} - t\right)^2}\right] \tag{13}$$

To calculate the areas of triangles S_{tr1} and S_{tr2}, values of l_1 and l_2 need to be obtained. The relationships between the central angles and l_1 and l_2 are:

$$l_1^2 = \left(\frac{D}{2}\right)^2 - \left(\frac{T}{2}\right)^2 \tag{14}$$

$$l_2^2 = \left(\frac{D}{2} - t\right)^2 - \left(\frac{T}{2}\right)^2 \tag{15}$$

Therefore, the triangles' area can be calculated by:

$$s_{tr1} = \frac{1}{2}\frac{T}{2} l_1 \tag{16}$$

$$s_{tr2} = \frac{1}{2}\frac{T}{2} l_2 \tag{17}$$

Rearranging above equations, the areal fraction of grain boundary and grain interior can be expressed respectively as:

$$A_G = \frac{\pi\left\{D^2 \cdot \cos^{-1}\left(1 - 2T^2D^{-2}\right) - (D-2t)^2 \cdot \cos^{-1}\left[1 - 2T^2(D-2t)^{-2}\right]\right\} + 360T\left[\sqrt{D^2 - T^2} - \sqrt{(D-2t)^2 - T^2}\right]}{T \cdot D/2} \tag{18}$$

$$A_{GB} = 1 - \frac{\pi\left\{D^2 \cdot \cos^{-1}\left(1 - 2T^2D^{-2}\right) - (D-2t)^2 \cdot \cos^{-1}\left[1 - 2T^2(D-2t)^{-2}\right]\right\} + 360T\left[\sqrt{D^2 - T^2} - \sqrt{(D-2t)^2 - T^2}\right]}{T \cdot D/2} \tag{19}$$

Substituting Eqs. (18) and (19) into Eq. (4), then the new constitutive model can be expressed by:

$$\sigma_y = \sigma_{f\mathrm{G}}\cdot\frac{\pi\left\{D^2\cdot\cos^{-1}\left(1-2T^2D^{-2}\right)-(D-2t)^2\cdot\cos^{-1}\left[1-2T^2(D-2t)^{-2}\right]\right\}+360T\left[\sqrt{D^2-T^2}-\sqrt{(D-2t)^2-T^2}\right]}{T\cdot D/2} + \sigma_{f\mathrm{GB}}\left\{1-\frac{\pi\left\{D^2\cdot\cos^{-1}\left(1-2T^2D^{-2}\right)-(D-2t)^2\cdot\cos^{-1}\left[1-2T^2(D-2t)^{-2}\right]\right\}+360T\left[\sqrt{D^2-T^2}-\sqrt{(D-2t)^2-T^2}\right]}{T\cdot D/2}\right\} \quad (20)$$

According to the previous studies [28], the relationship between average grain size D and the thickness t of grain boundary can be described as:

$$t = kD^n \quad (21)$$

where k and n are regarded as constants for a specific material. To copper and copper alloy, the correlation between grain size and thickness is:

$$t = 0.133D^{0.7} \quad (22)$$

In this way, there are only $\sigma_{f\mathrm{G}}$ and $\sigma_{f\mathrm{GB}}$ are unknown in Eq. (20) when σ_y is to be solved.

The flow stresses of grain interior and grain boundary of copper alloy are determined in the reference [19] and shown in Fig. 9.

It can be seen that the grain boundaries show a much faster rise in hardening rate compared to the gran interiors. This phenomenon has been exemplified by the results in the research of Hirth [33]. The grain boundary regions are shown with pronounced slip activity on two slip systems, which leads to a much higher hardening rate than that of the grain interiors.

Substituting the values of flow stresses of grain interior and boundary from Fig. 9 and Eq. (22) into Eq. (20), then the flow stress σ_y can be obtained. Figure 10 shows the comparison of the calculated and the experimental true stress-strain curves which were obtained under the condition of specimen's T/D >1. A good agreement between the calculations and experiments [34] illustrates that the developed constitutive model is capable to predict the relationship between stress and strain when the value of material's thickness/average grain size is less than 1 which can be frequent in microforming.

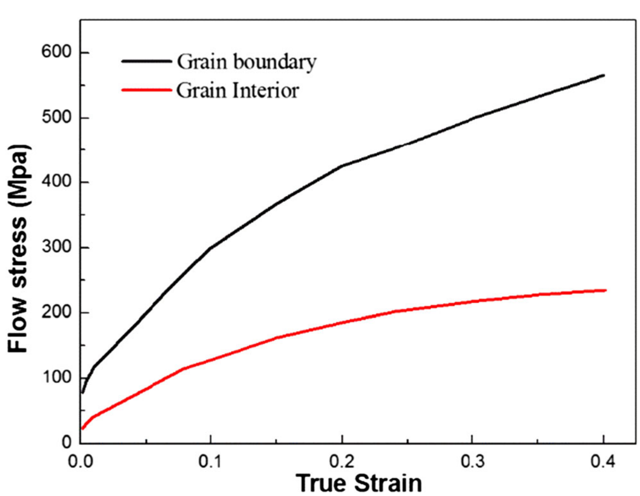

Fig. 9 Flow stresses of grain interior and boundary of pure copper and copper alloy sheet foil

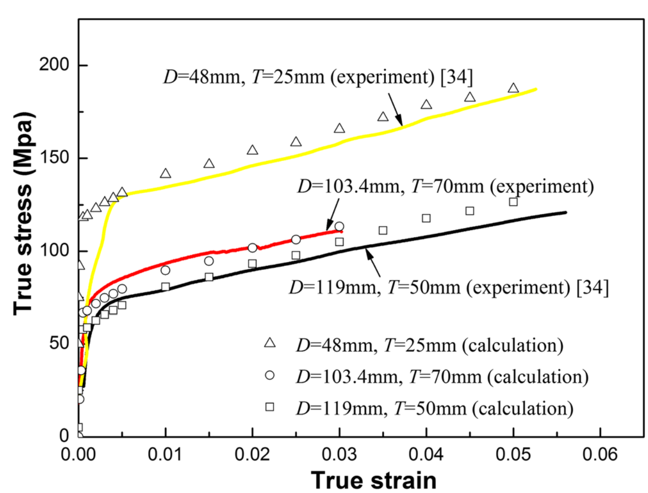

Fig. 10 Comparison of calculated and experimental stress-strain curves when T/D <1

5 Conclusions

In this paper, the influence of the ratio of thickness to average grain size on material's deformation and fracture behaviour was investigated. The ratios were chosen to be around 1, but in three different fields: *T*/*D* >1 (1.23), *T*/*D*≈1 (1.07) and *T*/*D* <1 (0.68). The following conclusions are obtained:

(1) Plastic deformation increase with the increase of *T*/*D*, and this is extremely obvious when comparing the plastic deformation which occurred with materials' *T*/*D* <1 to *T*/*D* >1. This shows that deformation behaviour is very sensitive to the value of the ratio of thickness to average grain size when the range is around 1.

(2) The number of micro-dimples decreases with the decrease of *T*/*D*, while the number of cleavage planes increases with it. When *T*/*D* >1, micro-dimple can be found are averagely distributed in fracture surface, which shows a great tendency of ductile fracture. However, a large area of cleavage plane means that brittle facture has a strong tendency to occur when *T*/*D* <1. Therefore, *T*/*D*≈1 can be regarded as the divide of ductile fracture and brittle fracture, and this ratio becomes a decisive factor to material's fracture mode.

(3) When *T*/*D* <1, there is only single incomplete grain in thickness direction, and classic surface layer model cannot be applied in this situation. A new constitutive model is developed based on composite model with the consideration of *T*/*D* <1. Comparison was made between simulation values and experimental ones, and a good agreement verified the validity of the developed model.

Acknowledgments The authors would like to thank the Australia Research Council (ARC) and Chinese Scholarship Council (CSC) for their financial support.

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