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Tensile deformation behaviors of pure nickel fine wire with a few grains across diameter

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Abstract: Size effects on plastic deformation behaviors in uniaxial micro tension of pure nickel fine wires were investigated experimentally, including flow stress and inhomogeneous deformation behaviors. It is found that with the increase of grain size or the decrease of number of grains across the diameter, the flow stress decreases and inhomogeneous deformation degree increases. When there are less than 9.3 grains across the diameter, the flow stress decreases quickly with the increase of grain size. Then, the flow stress size effect in micro tension of fine wires is revealed by a proposed model by introducing the grain boundary size factor. These results also indicate that both the fracture strain and stress decrease with the increase of grain size. When there are less than 14.7 grains across the diameter, both the fracture strain and stress decrease quickly. This indicates that the inhomogeneous deformation degree in micro tension increases with the decrease of the number of grains across the diameter. The fracture topography tends to be more and more irregular with the decrease of the number of grains across the diameter. Then, the formation mechanism of irregular fracture topography was analyzed considering the inhomogeneous distribution of microstructure when there are a few grains across the diameter.

Key words: nickel wire; size effect; micro tension; flow stress; inhomogeneous deformation; fracture

1 Introduction

With the rapid development of micro electro-mechanical-systems (MEMS) and micro system technology (MST), micro parts are widely applied in automotive, biomedical, and consumer electronics [1−3]. In the last two decades, microforming as a new micro manufacturing technology has played an important role in manufacturing micro metal parts. However, when dimensions of metal parts down scale to micro scale, the plastic deformation behaviors which dependent on the specimen size heavily are different from those in the macro scale, thus size effects occur [1].

KALS and ECKSTEIN [4] indicated that the flow stress decreases significantly with the decrease of specimen thickness by tension of thin sheet. GEIGER et al [1,5] proposed surface and mesoscopic models based on the fact that the free surface induced softening effects, and the reduction of flow stress in microforming was revealed. WANG et al [6] revealed the flow stress scatters in micro compression through the proposed model considering orientation distribution of the surface grain. LIN et al [7] proposed a size-dependant constitutive model based on the surface layer model by introducing the scale parameters and modifying the classical Hall−Petch equation to discuss the effect of the geometric dimensions and the grain size on the flow behavior in micro tension. ZHENG et al [8] performed uniaxial tensile tests of the pure Ti foils by a resistance-heating-assisted microforming process. A constitutive model based on the Fields−Bachofen (FB) equation which included the effect of the electrical current density on the work hardening and strain rate

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sensitivity was derived to describe the flow stress of ultrathin pure Ti under different forming conditions. KELLER et al [9,10] investigated the effect of the ratio of sheet thickness to grain size on the flow stress in tensile of pure Ni thin sheet. It is found that the flow stress tends to departure from the Hall−Petch relationship when there are less than about 4 grains across the thickness. CHEN and NGAN [11,12] found that the flow stress increases reversely with the decrease of wire diameter or the increase of grain size by tensile tests of silver wire with diameters in the range of 20 to 50 μm. This will happen for micron-sized specimens when the ratio of specimen diameter to grain size is smaller than a critical value of ~2 to 5. The TEM examination of the dislocation substructure suggests that the low ability for dislocations to accumulate inside the specimen is the reason for the existence of flow stress size effect. Apart from the flow stress size effect, the inhomogeneous deformation behaviors become distinct in micro scale. The surface topography tends to be rougher in micro compression [6] and the fracture strain decreases with the increase of grain size or the decrease of the specimen dimension [13−15]. CHAN et al [16] found that the degree of inhomogeneous deformation increases with the increase of grain size in micro-extrusion process of pure copper. KRISHNAN et al [17] found the extruded micro pins curve when using the coarse grained materials. CAO et al [18] proposed a model based on the crystal plasticity theory to reveal the curvature in micro-extrusion of coarse grained materials. WANG et al [19] investigated the effect of the ratio of cavity width to grain size on the filling behaviors through micro coining process. It is indicated that the filling ability is the worst when there are only about two grains across the cavity width at elevated temperature. WANG et al [20] found the similar results in micro coining at room temperature. WANG et al [21] indicated that the critical ratio is not only dependent on the ratio of cavity width to grain size, but also the cavity width.

In this work, micro tension tests of pure nickel fine wire with different grain sizes were carried out to investigate the effect of grain size on the deformation behaviors in the micro scale at room temperature. The effects of grain size on the flow stress, fracture strain, fracture stress and inhomogeneous deformation behaviors were discussed. The size effect on flow stress in micro tension of fine wires was revealed by a proposed model introducing the grain boundary size factor. The inhomogeneous deformation behavior resulting in the irregular fracture topography was analyzed considering the local microstructure distribution when there are a few grains across the diameter in micro tension.

2 Experimental

The raw material was cold-drawn pure nickel fine wires with purity of 98.5%. To reach various grain sizes, specimens were annealed in vacuum during 3 h at temperatures between 1073 and 1473 K and then air-cooled. The interactive effect of the specimen and grain sizes was quantified with the ratio of specimen diameter to grain size indicated by the ratio of specimen diameter to grain size (N). The microstructures of the heat-treated specimens by optical microscopy are shown in Fig. 1 and the obtained grain sizes, standard deviation (S_{dev}) of grain sizes and N are shown in Table 1.

Table 1 Mean grain sizes obtained under five different treatment conditions

Temperature/K	Grain size/μm	S_{dev}/μm	N
1073	18	9	55.6
1173	68	24	14.7
1273	107	35	9.3
1373	161	77	6.2
1473	288	68	3.5

Heat-treated pure nickel fine wires with 1.0 mm in diameter were selected for micro tension testing experiments. The tensile specimen gauge length was 100 mm according to the ASTM standard E8/E8M-13a [22]. In this research, five kinds of specimens were tested by an Instron testing machine with a load cell of 5 kN. The strain rate was constrained at a low level of 5×10^{-3} s^{-1} and the strain was measured by an extensometer mounted directly on the specimen gauge length. The specimens were strained till fracture. To increase the friction between the specimen surface and the fixture and avoid the fracture at the clamping positions, the clamping parts of the specimens were packaged by thermoplastic pipes under heating. The schematic pictures of micro tension of pure nickel fine wires and specimens before and after deformation are shown in Fig. 2.

3 Results and discussion

3.1 Flow stress size effect

Figure 3 shows the flow curves of specimens with different grain sizes. A decrease of flow stress occurs with the increase of grain size. The decrease of flow stress with grain size is distinct at lower strains and not distinct at higher strains. This is different from that in micro compression of cylinders with the same grain sizes [23,24]. These results are similar to those in tensile tests of foils [9,10]. To analyze the results in details, the relationship between inverse square grain size ($d^{-1/2}$) and

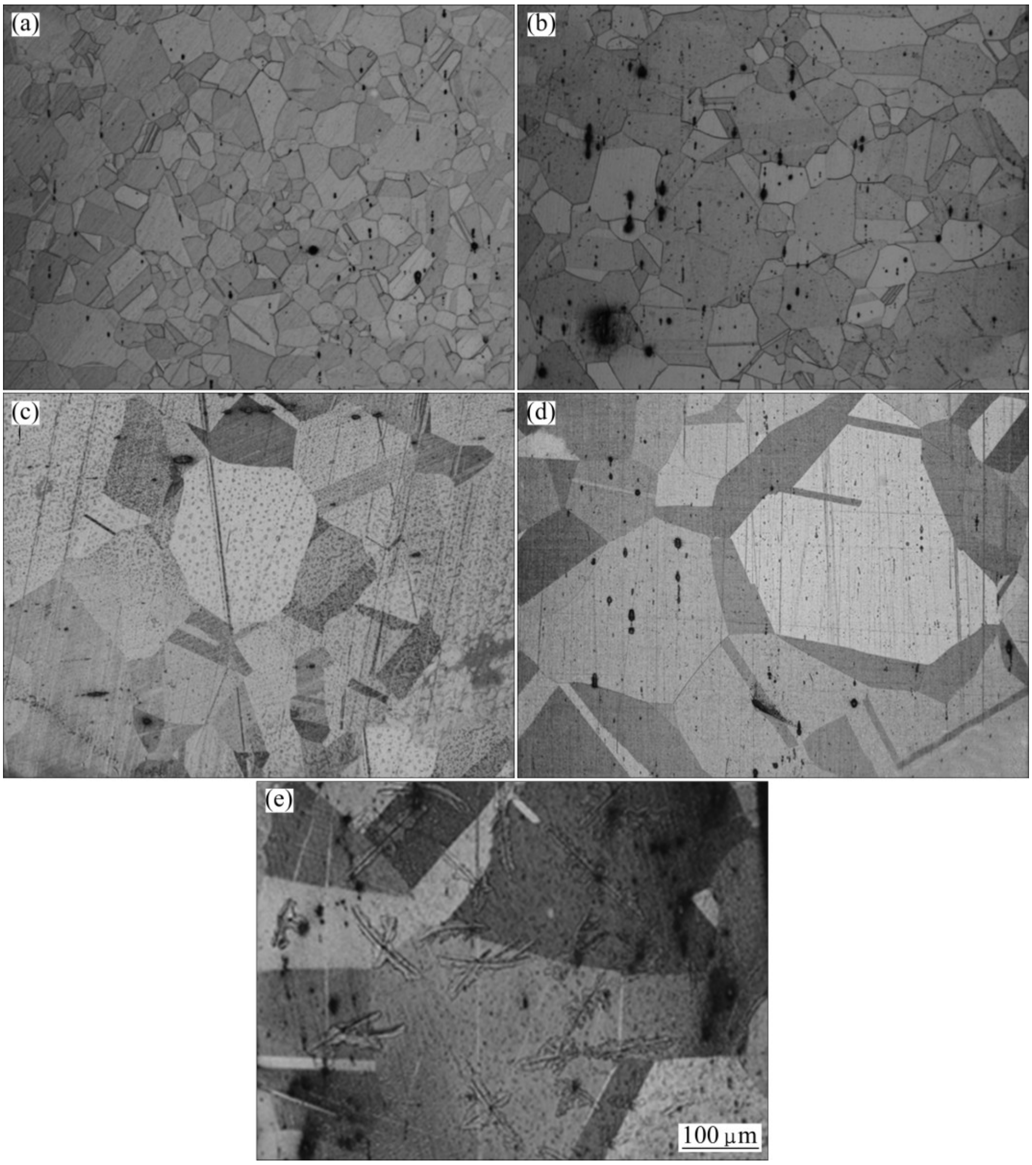

Fig. 1 Microstructures observed by optical microscopy in longitudinal cross-section of specimens with different grain sizes (d): (a) d=18 µm; (b) d=68 µm; (c) d=107 µm; (d) d=161 µm; (e) d=288 µm

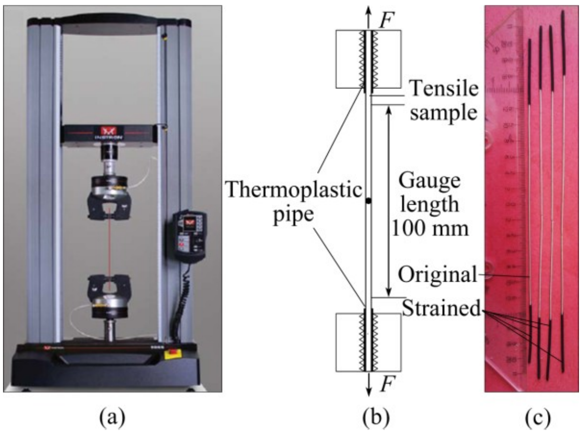

Fig. 2 Testing platform and scheme in micro tension of pure nickel fine wire: (a) Instron 5965Q; (b) Scheme of micro tension; (c) Specimens before and after deformation

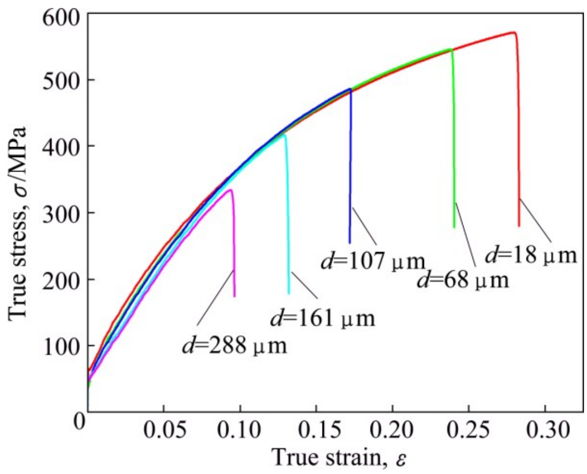

Fig. 3 Flow curves of specimens with different grain sizes in micro tension

true stress (σ) under different strains is depicted in Fig. 4. The strength of polycrystalline metals can be formulated by Hall−Petch relation which depends on grain size [25,26] and the grain size dependence can be formulated as follows:

$$\sigma(\varepsilon,d)=\sigma_0(\varepsilon)+\frac{K_{\mathrm{hp}}(\varepsilon)}{\sqrt{d}} \tag{1}$$

where $\sigma_0(\varepsilon)$ and $K_{\mathrm{hp}}(\varepsilon)$ are material constants at a given strain ε, d is average grain size, $K_{\mathrm{hp}}(\varepsilon)$ represents the grain boundary strengthening effect on the flow stress. The grain boundary density increases with the decrease of grain size, and specimen with smaller grain size results in a higher flow stress. MAHABUNPHACHAI and KOC [27] indicated that specimen size effect tends to play a significant role when there are 10−15 grains across the specimen size. In this work, for specimens with more than about 9.3 grains across specimen diameter, the true stresses under all strains are consistent with Hall−Petch equation, as shown in Fig. 4. Only grain size effect occurs in these situations. When there are less than 9.3 grains across the specimen diameter, the flow stress decreases quickly and departs from the Hall−Petch relationship. In this regime, the specimen size is expected to play an important role in the flow stress.

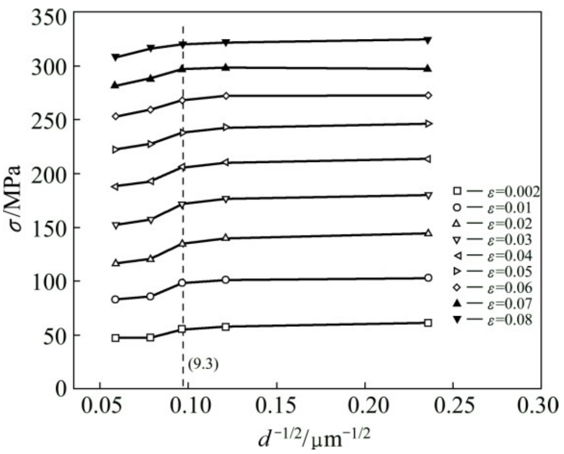

Fig. 4 Variation of flow stress (σ) with $d^{-1/2}$ under different strains in micro tension

To interpret the flow stress size effect on the ratio of specimen diameter to grain size, a grain boundary size factor η is introduced to characterize the effect of grain boundary on the mechanical property of polycrystalline materials in microforming. Thus, Eq. (1) is modified as follows:

$$\sigma(\varepsilon,d)=\sigma_0(\varepsilon)+\frac{\eta K_{\mathrm{hp}}(\varepsilon)}{\sqrt{d}} \tag{2}$$

The basic connotation of η is the relative length of grain boundary per unit area compared with the same grain sized specimen in macroforming. η equals 1 in macroforming and decreases with increase of grain size or the decrease of specimen diameter. To quantify η and simplify the problem, rectangular grains are applied to calculating the grain boundary size factor. The polycrystalline model is shown in Fig. 5. The grain boundary size factor is expressed as follows:

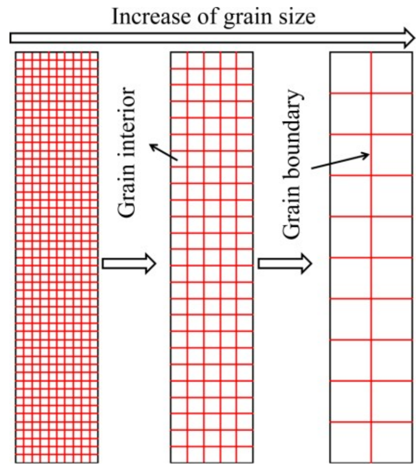

Fig. 5 Variation of grain boundary density with grain size in micro tension

$$\eta=1-\frac{d}{2D} \tag{3}$$

where D is the diameter of the pure nickel fine wire. For macroforming of polycrystalline materials, there are a large number of grains across the specimen diameter, i.e., η approaches 1. Equation (2) is translated into the Hall−Petch relationship for polycrystalline materials in macroforming. The variation of grain boundary size factor with ratio of specimen diameter to grain size for specimens with 1.0 mm in diameter is shown in Fig. 6. It is clearly indicated that the grain boundary size factor changes slightly when there are more than 10 grains across the specimen diameter. That can be regarded as that there is no specimen size effect. However, when there are less than 10 grains across the specimen diameter, the grain boundary size factor depends on the

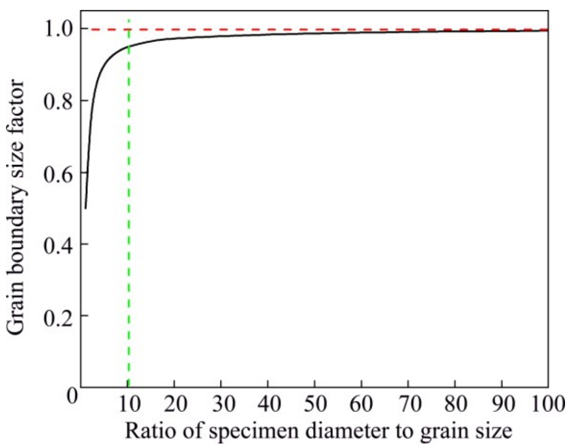

Fig. 6 Variation of grain boundary size factor with grain size

ratio of specimen diameter to grain size strongly, and thus the size effect of the specimen occurs. The critical ratios calculated by the proposed model and in the experiment are close.

Then, the valid of the proposed model is evaluated as follows. Two parameters of $\sigma_0(\varepsilon)$ and $K_{hp}(\varepsilon)$ are only related to the materials and strains, not dependent on the grain size or specimen size. Thus, the two parameters in Eq. (2) can be obtained from the flow curves of specimens with grain sizes of 18 and 68 μm by combining with Eq. (3), as shown in Fig. 7. $\sigma_0(\varepsilon)$ increases with strain linearly. $K_{hp}(\varepsilon)$ is not dependent on the strain in the range of strain from 0.002 to 0.08. Thus, the flow stresses of specimens with grain sizes of 107, 161 and 288 μm can be calculated by combining Eqs. (2) and (3) and the two parameters obtained above. Then, the variation of calculated true stresses and $d^{-1/2}$ under strains (0.002−0.08) is shown in Fig. 8. The critical ratio for specimen size effect of flow stress is close to that in the experiment. The experiment results and calculated ones match well. Thus, the proposed model can be used to model the specimen size effect of flow stress when there are only a few grains across the diameter.

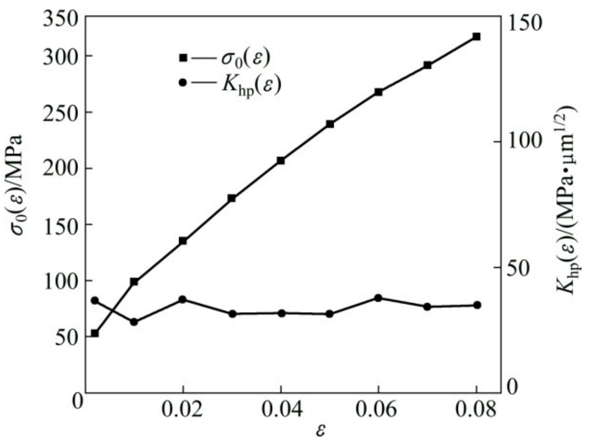

Fig. 7 Variations of $\sigma_0(\varepsilon)$ and $K_{hp}(\varepsilon)$ with strain

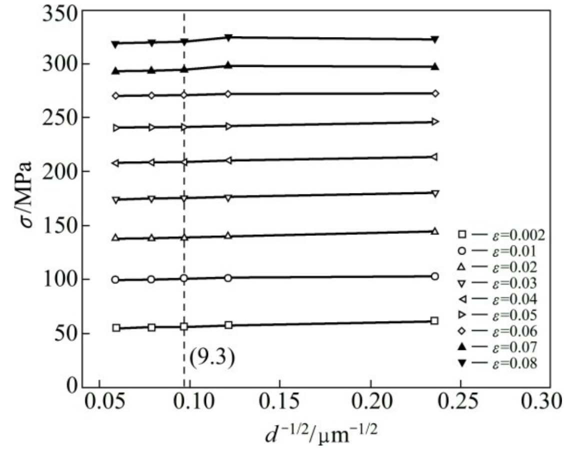

Fig. 8 Variation of flow stress with $d^{-1/2}$ under different strains in micro tension by proposed model

Figures 9 and 10 show the fracture strain and stress of specimens with grain size, respectively. It is indicated that both the fracture strain and stress decrease with the increase of grain size or the decrease of the ratio of specimen diameter to grain size. For face-centered cubic (FCC) metals such as pure nickel, the main deformation mode is dislocation slip. The more the slip systems are, the better the ductility is. Generally, specimens with smaller grain sizes or more grains result in a better ductility because of more slip systems participating to coordinate intergranular deformation. The deformation compatibility is better when the microstructure is homogeneous. Thus, the necking generally occurs at the location with inhomogeneous distribution of microstructure. When there are a few grains across the specimen size as shown in Fig. 1 and Table 1, the inhomogeneous distribution of grain size tends to increase with the increase of grain size. When there are fewer grains across the specimen diameter, at some cross-sections of the specimen, the microstructure distribution is more inhomogeneous than that of the mean distribution of the whole specimen. The increase of inhomogeneous distribution of microstructure results in the occurrence of necking earlier in micro tension when there are fewer grains across the specimen diameter. The fracture strain decreases as the deformation can be

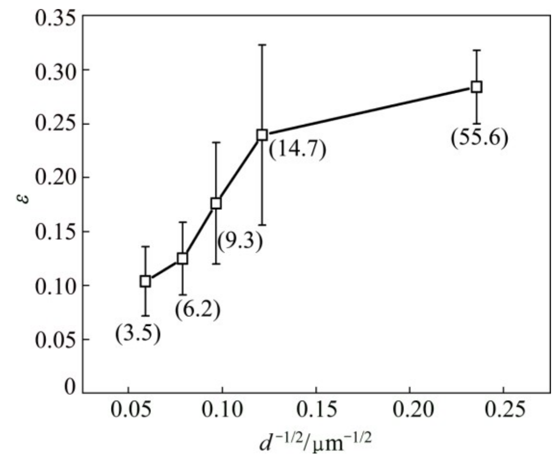

Fig. 9 Variation of fracture strain ε with $d^{-1/2}$

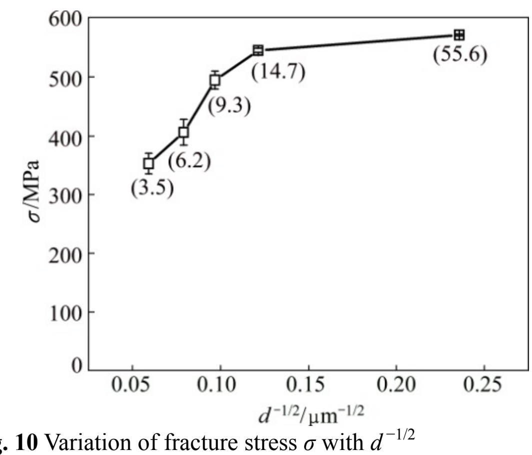

Fig. 10 Variation of fracture stress σ with $d^{-1/2}$

localized at the grains which are favorable to the deformation in the tensile direction. Accordingly, the fracture strain and stress decrease with the increase of grain size or the decrease of the ratio of specimen diameter to grain size. And the critical ratio of specimen diameter to grain size for the fracture strain and stress is larger than that for flow stress. Because the flow stress depends on the whole specimen microstructure, the fracture strain and stress mainly depend on the local distribution of the specimen microstructure.

3.2 Inhomogeneous deformation behaviors

Figure 11 shows the fracture micro topographies of different grain sized specimens in micro tension. There are many dimples with micro voids in the fracture topographies. The fracture mode is ductile fracture. The size, depth and distributions of dimples tend to be irregular with the decrease of ratio of specimen diameter to grain size. These inhomogeneous distributions of dimples result from the inhomogeneous distribution of material microstructure in the deformation region. Figure 12 shows the macro topographies of necking regions of different grain sized specimens in micro tension. It is indicated that there are many slip steps along the loading direction. The shape of the necking region is conical and the taper angle tends to increase

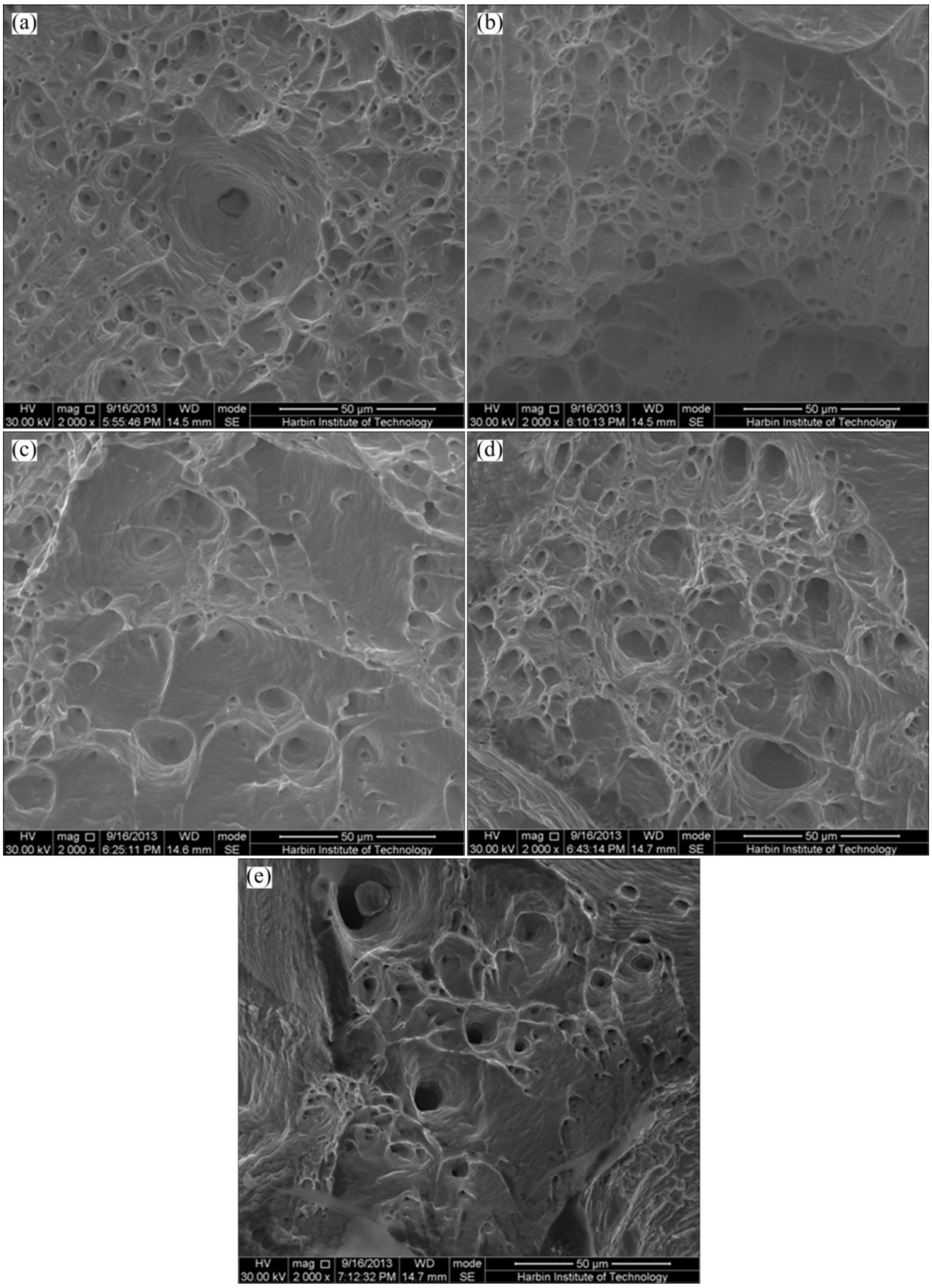

Fig. 11 Fracture micro topographies of specimens with different grain sizes: (a) d=18 μm; (b) d=68 μm; (c) d=107 μm; (d) d=161 μm; (e) d=288 μm

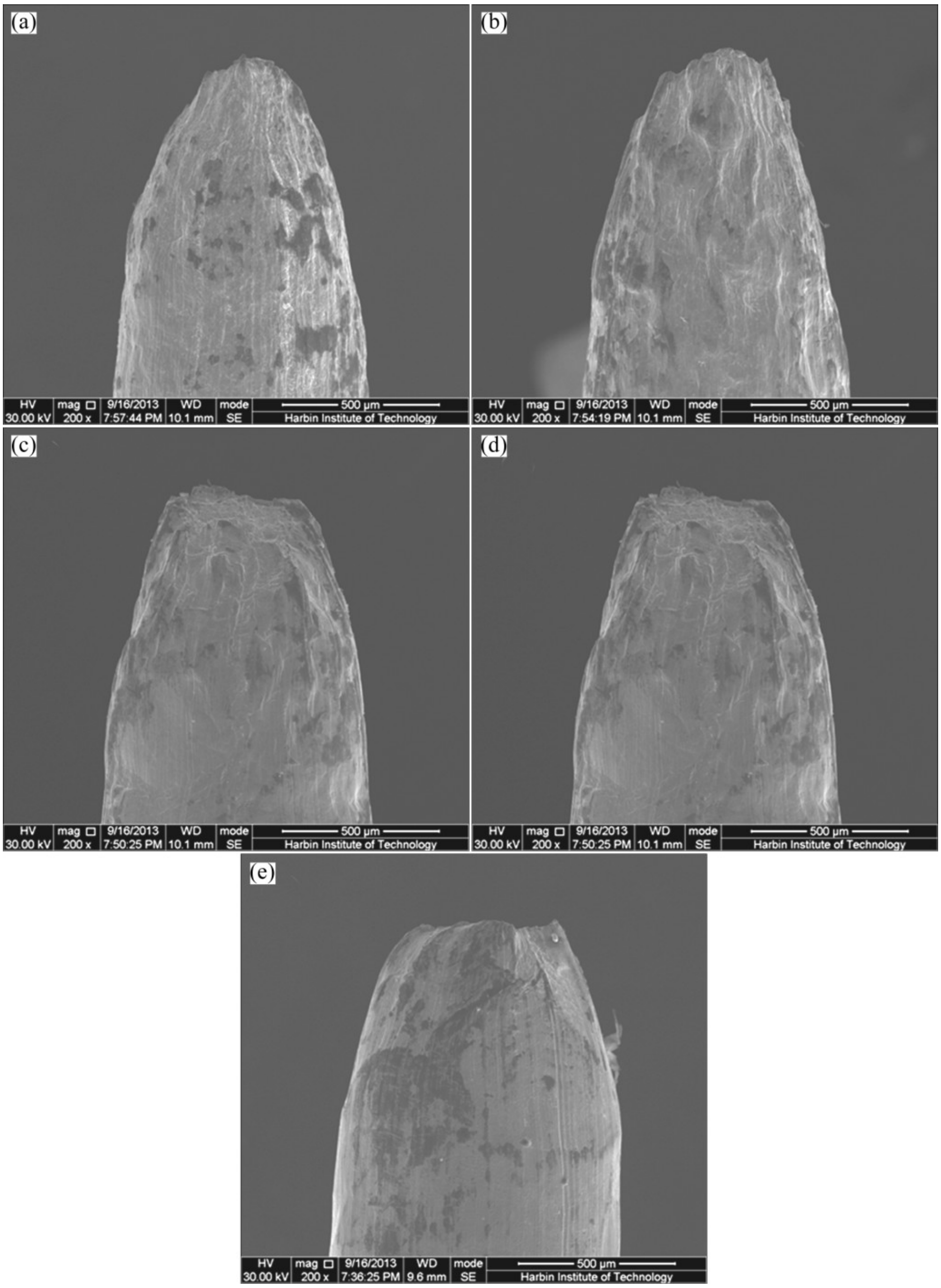

Fig. 12 SEM images of necking regions of specimens with different grain sizes in micro tension: (a) d=18 μm; (b) d=68 μm; (c) d=107 μm; (d) d=161 μm; (e) d=288 μm

with the decrease of ratio of specimen diameter to grain size. These indicate that the smaller the taper angle is, the better the ductility is. The ductility decreases with the increase of grain size or the decrease of ratio of specimen diameter to grain size.

Figure 13 shows the whole topographies of the fracture sections of different grain sized specimens in micro tension. It is indicated that the shapes of fracture sections are changed from regular circle to irregular polygon with the decrease of ratio of specimen diameter to grain size. For metal crystalline, the deformation resistance of grain boundary is larger than that of inner part of grain. The surface grains with free surfaces are deformed normally to the free surface easily in the deformation. This results in the inhomogeneous deformation of grains with free surfaces. Deformation behaviors of every single grain are related to its position, orientation, stress status and surroundings. Thus, the deformation behaviors of every grain are different. With the increase of grain size, the number of grains across the diameter decreases. The deformation behavior of every single grain plays more and more important role in the

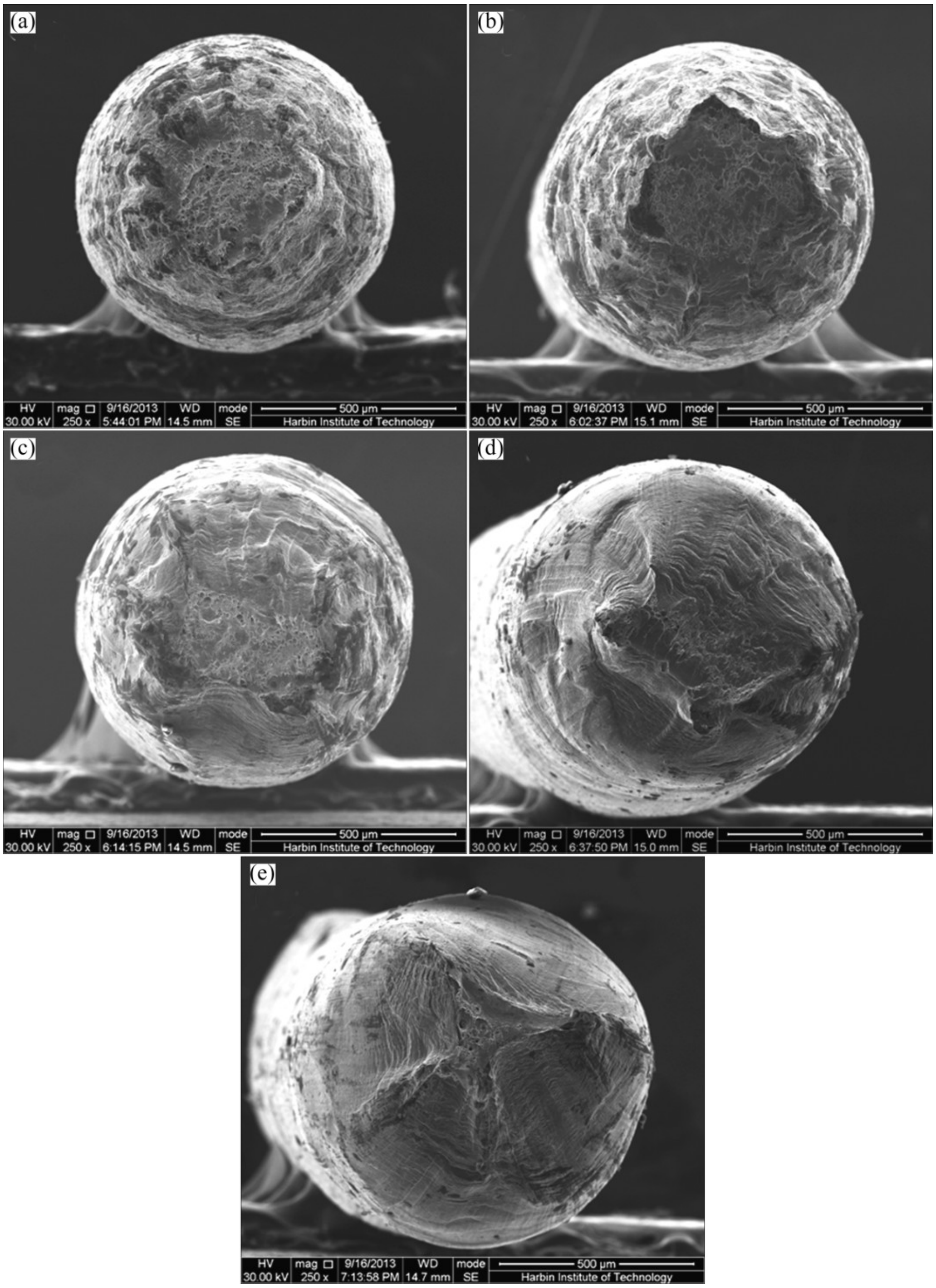

Fig. 13 Whole fracture topographies of specimens with different grain sizes in micro tension: (a) d=18 μm; (b) d=68 μm; (c) d=107 μm; (d) d=161 μm; (e) d=288 μm

overall deformation behaviors of the specimens. When there are only fewer grains across the diameter, the microstructure distribution is very inhomogeneous and random, and the strain incompatibility among the finite number of grains causes severe local deformation in some grains. Surface grain interiors with different orientations flow normally to the surface inward and outward under axial loading in micro tension. Convex and pit are formed on the specimen surface, and the sharp corners are formed at the grain boundaries in general. When the specimen diameter is much larger than the grain size, the sizes of convex and pit are so small to be seen obviously compared with the contour dimension of the fracture section, as shown in Fig. 13(a). However, when there are only a few grains across the specimen diameter, inner parts of two adjacent grain flow towards the specimen surface, convex or pit are formed on the specimen surface, and a sharp corner is formed at the grain boundary between two adjacent grains, as shown in Fig. 13(e).

4 Conclusions

1) The specimen size effect on flow stress occurs when there are less than about 9.3 grains across the specimen diameter. The specimen size effect on flow stress is revealed by the proposed model by introducing the grain boundary size factor.

2) The fracture strain and stress decrease with the increase of grain size or the decrease of ratio of specimen diameter to grain size, which result from the ratio of specimen diameter to grain size and the inhomogeneous distribution of microstructure with the increase of the grain size.

3) The fracture section contour tends to be more and more irregular with the decrease of the number of grains across the diameter and the formation mechanism is revealed considering the inhomogeneous distribution of microstructure.

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直径方向上仅有几个晶粒时纯镍丝材的拉伸变形行为

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摘 要：研究纯镍丝材单向微拉伸塑性变形过程中的流动应力和非均匀变形行为尺寸效应。实验发现，流动应力随晶粒尺寸的增加(或直径方向上晶粒数量的减少)而降低，而非均匀变形程度增加。当直径方向上少于 9.3 个晶粒时，流动应力随晶粒尺寸增加而快速降低。通过引入晶界尺寸因子构建介观尺度材料本构模型揭示丝材微拉伸流动应力尺寸效应。结果表明，断裂应变和断裂应力随着晶粒尺寸的增加而减小。当试样直径方向上少于 14.7 个晶粒时，断裂应变和断裂应力快速降低，表明微拉伸过程中的非均匀变形程度随着直径方向上晶粒数量的减小而增加。当试样直径方向上的晶粒数量减少时，断口形貌变得越来越不规则。从材料微观组织分布方面分析了不规则断口形貌的形成机理。

关键词：镍丝；尺寸效应；微拉伸；流动应力；非均匀变形；断裂

(Edited by Wei-ping CHEN)