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ARTICLE

Deformation Behavior of Pure Tungsten During Equal Channel Double Angular Pressing

Li Ping, Duan Zihao, Wu Tao, Hua Yaling, Xue Kemin

Hefei University of Technology, Hefei 230009, China

Abstract: As a new severe plastic deformation (SPD) method, equal channel double angular pressing (ECDAP) is developed based on equal channel angular pressing (ECAP) to reduce the offset load. The deformation behavior of commercial sintered pure tungsten (W) during single pass ECDAP was analyzed quantitatively in this paper by finite element simulation and experiment investigation. The results show that, compared with ECAP, ECDAP exhibits more homogeneous local force of the die and improves the offset load of the punch with the same parameters. Meanwhile, the average effective strain introduced by ECDAP is similar to that of ECAP. Nevertheless, the uniformity of strain distribution is also greatly improved owing to the secondary shear deformation. In addition, grain size decreases to 1.77 μm in the secondary shear deformation zone and the grains are elongated obviously in the angle zone. Hence, the microhardness is enhanced because of the Hall-Petch strengthening and strain strengthening. These results indicate that the ECDAP process is a promising approach for producing ultra-fine grains metallic materials.

Key words: pure tungsten; ECDAP; the secondary shear; grain refinement; microhardness

As refractory rare metal, tungsten (W) is well-known for its excellent physical and chemical properties, such as high melting point, high strength, high sputtering resistance and low deuterium/tritium retention. Owing to these, W has wide application prospect in aerospace, electronics, chemical, nuclear industry and other extreme environment. However, the further application of W is limited due to its low temperature brittleness, recrystallization brittleness, etc. As we all know, grain size is a very important microstructural factor affecting almost all aspects of polycrystalline or single crystal metals. Ultrafine grained (UFG) materials demonstrate many unusual features of mechanical behavior, Such as high yield strength, low strain hardening and specific fatigue behavior [1]. As a result, UFG has gained more and more attentions in the field of material preparation and properties. For example, UFG and nanostructured (NC) materials have better plasticity, strength and ductility in comparison with conventional coarse-grained (CG) materials [2]. For refractory metals, the UFG or NC W can not only improve the strength and toughness of the material but also improve the ductility at low temperature [3]. Therefore, refining grains and optimizing microstructure is the key to promote the development of W and expand its applications. Currently, a lot of experimental researches show that the UFG or NC microstructures can be obtained through different technologies. For example, Zhang et al [4] prepared UFG copper by the SPS process with reasonable parameters. Okitsu et al[5] investigated that the UFG ferrite was obtained when a low carbon steel was cold-rolled to 91% reduction and then annealed at 893~928 K.

Severe plastic deformation (SPD) has great potential to refine the grain size of initial CG metals significantly, which has been gradually applied to improve the microstructure of tungsten and its alloys. Valiev[6] demonstrated that metals and alloys with mean grain size of 100 nm or less could be obtained by a SPD deformation. From then on, UFG of different materials were prepared by various SPD processes. Wei et al [7] stated that NC W was obtained by high-pressure torsion (HPT) with extremely high strength, and the ductility was also enhanced. Nevertheless, it is worth noting that HPT cannot fabricate large size ultrafine-grained samples. Compared with HPT, the equal channel angular pressing (ECAP) is an important SPD technology to fabricate large

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Corresponding author: Xue Kemin, Professor, School of Materials Science and Engineering, Hefei University of Technology, Hefei 230009, P. R. China, Tel: 0086-551-62901368, E-mail: xuekm@hfut.edu.cn

bulk UFG metals, in which specimens are squeezed into a specially designed die to achieve large shear deformation. Fig. 1a shows the schematic diagram of ECAP equipment with an inner angle (Φ) and an outer angle (ψ). A simple shear strain is introduced when a sample passes through the intersection of the two channels. Kim et al [8] summarized that the value of shear strain relies on geometry of the die, which is defined by Φ, ψ and the radius of the angle of the die. In general, Φ of the die is about 120° for the present ECAP experiments; However, the strain of the single pass deformation is so small. As we can see from Fig. 1a, the cross-sectional dimensions of the sample remain unchanged, and thus strain can be accumulated by multi-pass ECAP. Cirri et al [9] has shown that grain refinement can be achieved by multi-pass ECAP deformation. Via this technology, the relative density and microhardness uniformity of pure molybdenum powder were improved, and the grain size was significantly refined [10]. For this process, if the single-pass strain value is small, it is not conducive to the formation of dislocations, sub-grains and large-angle grain boundaries, so the improvement of material performance will be limited.

Equal channel double angular pressing (ECDAP) process is proposed which based on ECAP with Φ divided into two continuous angles (Φ_1, Φ_2) in Fig.1b. During ECDAP, a sample is squeezed into a die consisting of three channels with equal cross-section intersect at two angles Φ_1, Φ_2. Owing to two shear deformations within one extrusion, strain accumulation can be achieved. We have performed numerical simulation analysis on ECDAP process (as in section 3.2). Compared with ECAP (Φ = 90°), the stress distributed on the punch is more even, and the wear is less on the inner angle of ECDAP die. Compared with ECAP (Φ = 135°), the coefficient of variation of the sample is relatively low after four passes ECDAP, which means that the strain is more uniform after ECDAP deformation. Therefore, the ECDAP has great advantage on improving the strain uniformity of the sample. In addition, ECDAP can also improve unbalance loading and wearing, thus prolonging the life of dies, so as to reduce experimental cost.

In this paper, the pure W was deformed by ECDAP through the method of combining numerical simulation and experiment investigation. Then, the stress and strain behavior, distribution of grain size and microhardness were quantitatively analyzed. Meanwhile, the grains refinement and strengthening mechanism were discussed briefly. This work provides a comprehensive understanding of microstructure evolution and mechanical properties during ECDAP and provides supports to the application of SPD techniques in materials modification.

1 Modeling Procedures

The sandwich mold structure was designed and the corresponding finite element model for ECDAP process is shown in Fig.2. The cross section of the vertical and horizontal channel is 20 mm×20 mm, and a 16 mm transition oblique channel with the same cross section is added between the two channels. Two inner angles (Φ_1 and Φ_2) with the same value of 135° are formed without outer angle, but the two angle (Ψ_1, Ψ_2) are formed. The dimension of the sample is 20 mm×20 mm×60 mm, which was divided into 50000 tetrahedron grid elements in the software of DEFORM-3D. The stress-strain curves of pure W used for ECDAP was established as shown in Fig. 3, which was based on the hot compression experiments at different strain rates. According to the temperature range for plastic deformation of W and the experiment condition, the sample and ECDAP die temperature were set as 1373 and 873 K, respectively. The punch moved at a constant speed of 2 $mm\cdot s^{-1}$ and the factor of friction between the sample and the die was set as 0.1 because the process is conducted under well-lubricated conditions. In this way, the simulation results of single-pass ECDAP process of pure W was obtained. The X, Y and Z axis were parallel to extrusion direction, vertical direction, and transversal direction, respectively.

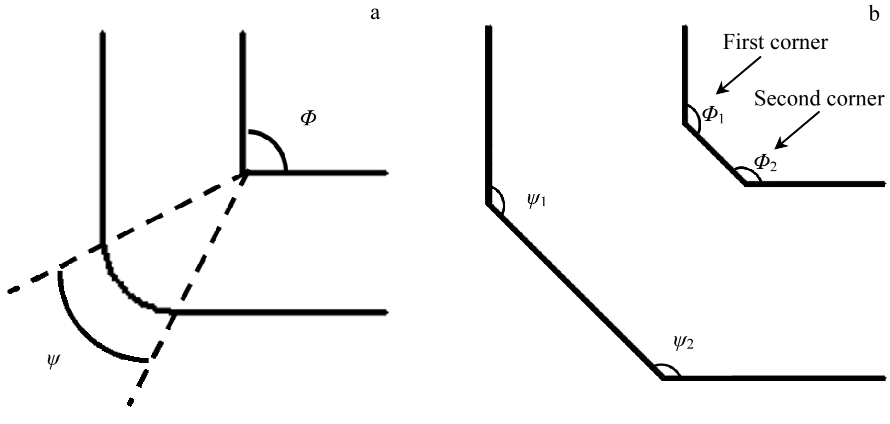

Fig.1 Schematic illustrations of ECAP (a) and ECDAP (b)

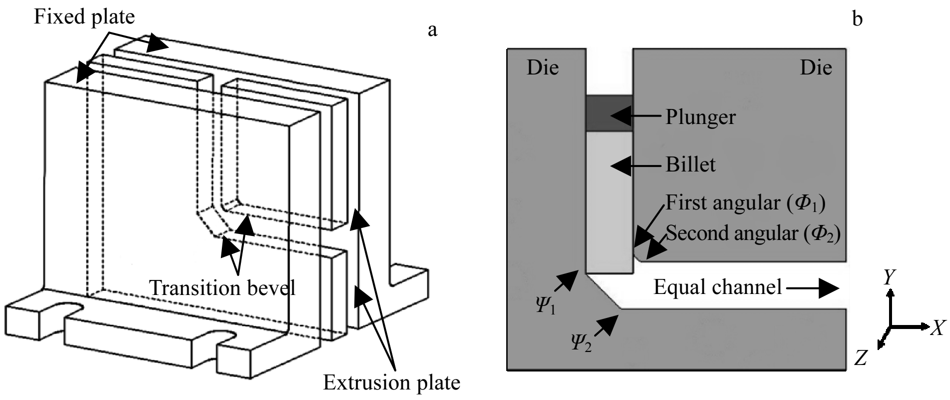

Fig.2 Schematic illustrations of the ECDAP die structure (a) and die slicing (b)

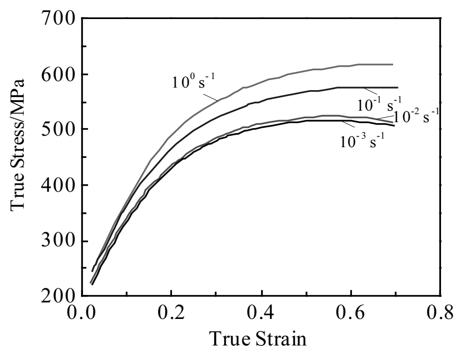

Fig.3 True stress-strain curves of W obtained from hot compression tests at 1173 K with different strain rates

2 Experiment

Commercially pure W rod with a purity level of 99.9% was used for this investigation. Nominal composition of the initial W is listed in Table 1. The sample with the dimension of 19.5 mm ×19.5 mm ×60 mm was prepared by a wire-cutting electric discharge machine. Then the high temperature lubricants (NNT LRA-15 Boron Nitride) were used to decrease the friction between the mold cavity and the sample. The experiment was carried out on a RZU200HF plastic forming machine. Prior to ECDAP, the sample was first heated in a furnace at 1373 K and incubated for 30 min; simultaneously the mold was heated to 873 K by a resistant heating ring. Following ECDAP, the *X* and *Z* planes of the shear zone (Fig.2b) were mechanically polished to a mirror-like surface using different grit of SiC paper starting from 200# to 2000# grit for the mechanical and microstructural characterization. In advance of microstructure observation, the mechanically polished samples were subjected to corroding with a solution of 10 mL $NH_3{\cdot}H_2O$, 30 mL H_2O_2 for 3 min to 4 min. Subsequently, the morphology was observed by MR2000 optical microscopy. The microhardness was measured on the polished *X* plane via a MH-3L Vickers microhardness tester, and ten measuring points along the normal direction in the shear deformation zone were made for each sample. For each microhardness measurement, the applied indenting load and dwell time were 300 g and 15 s, respectively.

3 Analysis and Discussion

3.1 Simulation accuracy analysis

In order to effectively and intuitively illustrate the accuracy of the simulation, the simulated grid is coincident with the physical map, and the results are shown in Fig.4. The physical map has a good coincidence with the simulated grid, and there is a slight difference between the head and the part of the sample because a large amount of flash is generated during the experiment, so that the metal passing through the passage is reduced. Then the width of the sample and the simulated grid sample at different positions of the sample are measured. The measurement position is shown in Fig.5, and the results show that the sample has a high degree of coincidence with the simulated grid.

3.2 Load analysis

Fig.6 shows the load-stroke variation and change of the grids during the single pass ECDAP deformation of pure W. According to the numerical simulation results, the ECDAP can be divided into three stages, (I) primary shear deformation stage, (II) secondary shear deformation stage (shear deformation through the second angle) and (III) steady

Table 1 Nominal composition of pure tungsten (wt%)

W	O	C	Mo	V	Co	Ni	P	Others
>99.9	<0.04	<0.003	<0.003	<0.001	<0.005	<0.001	<0.0008	<0.022

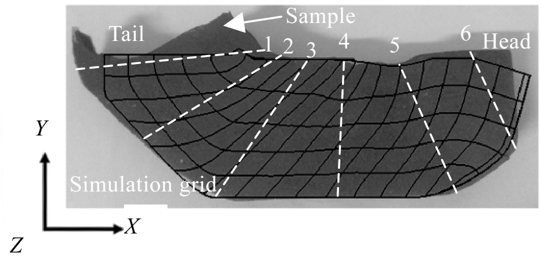

Fig.4 Sample and simulation grid

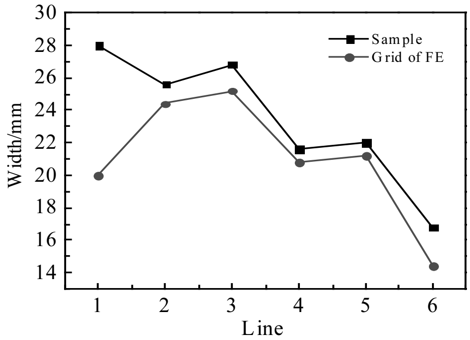

Fig.5 Physical and simulated grid widths at different locations

deformation stage. The load was increasing when the head of sample entered the horizontal channel without shear deformation. Subsequently, the metal gradually flowed and underwent shear deformation at the first 135° angle Φ_1, and then a shear zone was formed. The load was approximately stable. With the development of the deformation, the sample was contacted with the angle Ψ_2, resulting in a rapid increase of the load. And then the sample underwent the second shear deformation after it entered the second 135° angle Φ_2, and the load was stable with a slight decline. As the deformation further progressed, the sample was extruded into the horizontal channel of the die and the load increased slowly due to the friction effect. Then the sample was in a stable deformation stage subjected to shear deformation and friction, and the load tended to be steady. As we can see from Fig.6, the square grids became parallelogram grids after they passed through the double corner, which indicated that the specimen was subjected to shear deformation in the angle zone.

Die stress distribution during ECAP (the simulation parameters and process settings were consistent with ECDAP) and ECDAP single-pass deformation is visible in Fig.7. It is clearly observed that there are stress gradients, especially for ECAP. The stress distribution is mainly in the angle zone, which is much bigger than that of other zones of die. However, the greater stress value means greater damage to the die, resulting in reduction of die life. It is noticeable that the stress distribution on the punch of both dies is not symmetrical; therefore, 20 points were selected to evaluate the stress uniformity of each punch. From the Fig.8, we can know that there is a big difference between maximum and minimum stress on the ECAP punch, while it was reduced by ECDAP, and uniformity of stress distribution on the punch was also improved. In summary, ECDAP improved the stress state of the die and reduced the offset load of the punch, which is conducive to the deformation of high strength and refractory metals.

3.3 Strain analysis

The strain distribution after single pass ECDAP and ECAP are shown in Fig.9. The samples can be separated into three parts: (Ⅰ) tail, (Ⅱ) body and (Ⅲ) head; what's more, the body processed by ECDAP includes the primary shear zone and secondary shear zone. For ECDAP, the volume fraction of body is about 90% of total sample, while the tail and head zone are small, which are better than that processed by ECAP. Meanwhile, the secondary shear zone accounts for more than 60% of total sample, where the effective strain is higher than that of other zones with the value more than 1. Comparing the two samples, we can know that effective strain of the sample of ECDAP is 0.952, and the strain distribution in shear zone is uniform, which are also better than that of ECAP. In brief, strain uniformity of the sample processed by ECDAP was improved enormously.

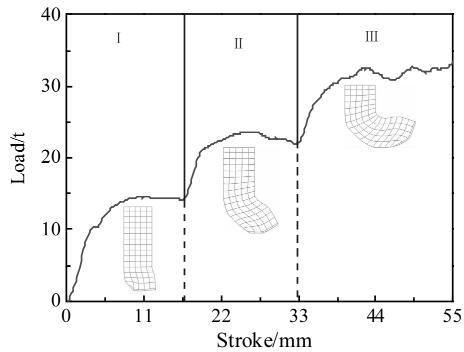

Fig.6 Load-stroke and grids variation in different stages

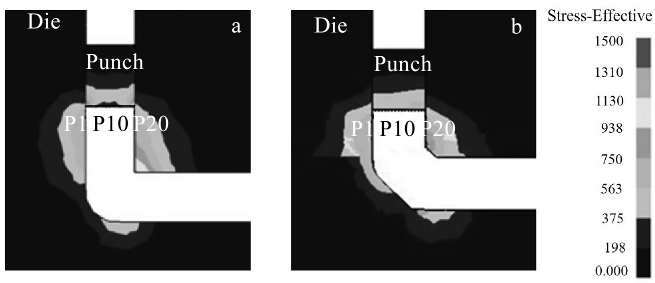

Fig.7 Die stress distribution of single-pass deformation of pure W: (a) ECAP and (b) ECDAP

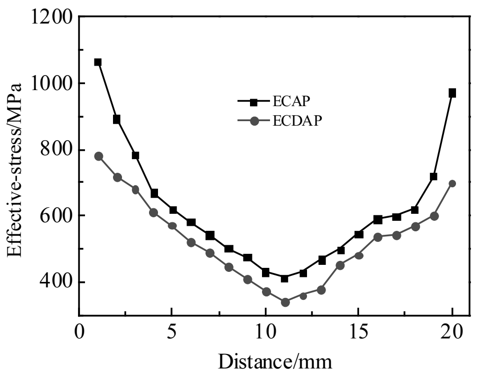

Fig.8 Stress value distribution on punch

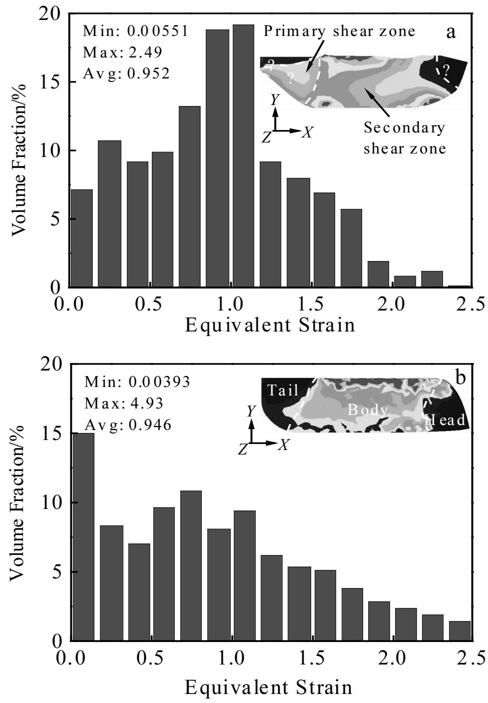

Fig.9 Effective strain distribution and percentage of single pass deformation: (a) ECDAP and (b) ECAP

Simultaneously, it is demonstrated that the double angle structure of ECDAP can achieve the large strain accumulation by two shear deformations during one extrusion.

ECAP is also a method for fabricating bulk ultrafine grain material by simple shear deformation, which refers to uniform plane rotation deformation.

Fig.10a shows the schematic diagram of a simple shear model. The shear zone is mainly affected by shear force, and the plane grid of the primary shear zone and the secondary shear zone were twisted along the *Y* and *X* axis, respectively. As shown in Fig.10b, an initial grid EFGH is marked in black, $E_1F_1G_1H_1$ and $E_2F_2G_2H_2$ are grids after the primary and secondary shear deformation, respectively. The shear angle α of the grid in the primary shear zone is about 34°. Its shear strain can be calculated as tangent of shear angle by the following Eq. (1) which is shown in Segal's work [11].

$$\gamma = \frac{\mathrm{tg}\varphi}{\sqrt{3}} \tag{1}$$

where γ is shear strain, and φ is shear angle of grid.

During the second shearing process, the reference axis of the grid shear angle converted to *X*-axis; therefore, its shear angle β of the grid is about 59°. And the shear strain can be calculated by following modified Eq. (2).

$$\gamma = \frac{-\mathrm{tg}(\varphi + \pi/2)}{\sqrt{3}} \tag{2}$$

Correspondingly, the shear strain of the primary shear zone and the secondary shear zone were about 0.39 and 0.96, respectively. The shear strain in the secondary shear zone is quite large and approaches the average effective strain (the value is 0.952) of the sample. This illustrates that the secondary shear is very effective in accumulating large strain.

In order to study the strain variation in different zones in detail, seven tracking points were selected on the *Y* and *Z* intermediate planes, which are depicted in Fig.11a. And the effective strain of each point versus time is shown in Fig.11b. During ECDAP, effective strain of each point accumulates continuously and saturates to a maximum value finally, but the value of each point is different. For example, P5 is located at the head of the sample. Without

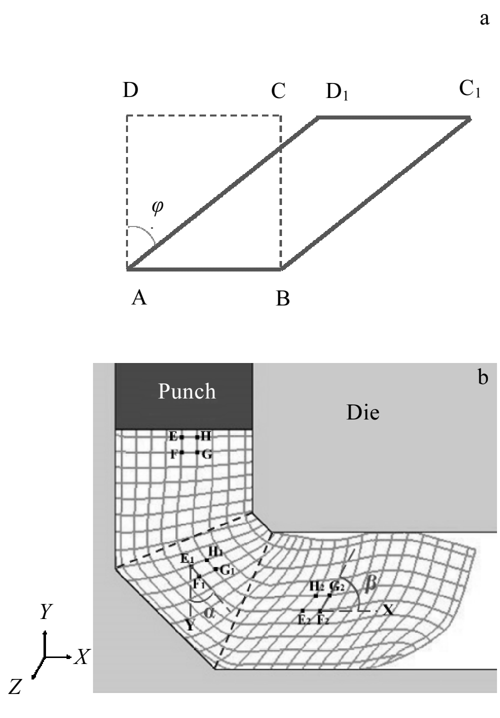

Fig.10 Simple shear model (a) and grid distribution during ECDAP deformation (b)

shear deformation, its effective strain increases dramatically at first and then slowly. P1 is located at the center of the sample, and has undergone two shear deformations, hence, its effective strain increases gradually with the deformation process. P4 is located at the tail of the sample, and it does not enter the shear zone of the mold, so its effective strain accumulation is relatively smaller. The strain evolutions of other points (P2, P3, P6 and P7) are consistent with P1. However, due to the friction effect, the strain accumulation of each point is quite different after ECDAP, for instance, effective strain of P3, P2 are larger than that of P6 and P7. The strain value of each point represents the deformation in different zones. Therefore, it can be concluded that, for the whole sample, the degree of metal deformation at the inner and outer angle is large than that of other zones; moreover, the strain of inner angle is larger than that of outer angle.

3.4 Microstructural analysis

Fig.12a displays the microstructure of the initial W. It is shown that the grain size is in the range of 5 ~ 20 μm, with an average one of 10 μm. The shape of grain is basically irregular. Fig.12b~12g show the microstructure of *X* plane of the shear zone processed by single pass ECDAP. It can be seen that the grains of the shear zone are more equiaxed and homogeneous. The grain sizes of the primary and secondary shear zone are mainly in the range of 2.41~4.73 μm and 1.77~2.32 μm, respectively. The refinement of grain and distribution homogeneity in the secondary shear zone are both higher than those in the primary shear zone. It indicates that the shear strain can be greatly enhanced by the secondary shear deformation, and the double angles structure has obvious effect on grain refinement during extrusion deformation. In particular, comparing Fig. 12b with 12e (taken from inner angle zone of the primary shear

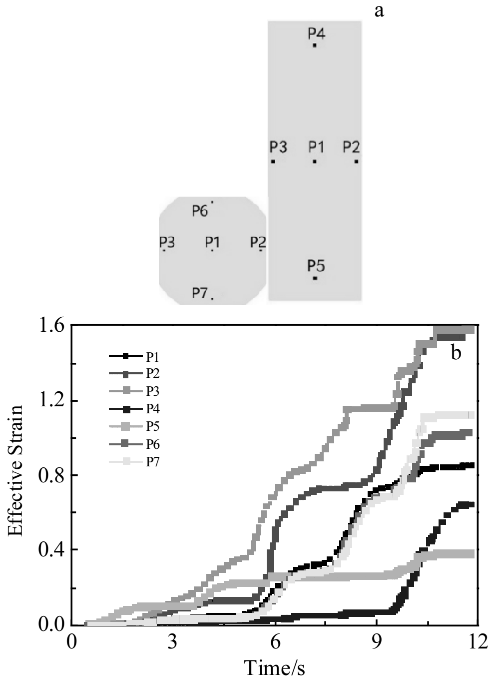

Fig.11 Position on the *Y* and *Z* intermediate planes (a) and various effective strain of tracking points (b)

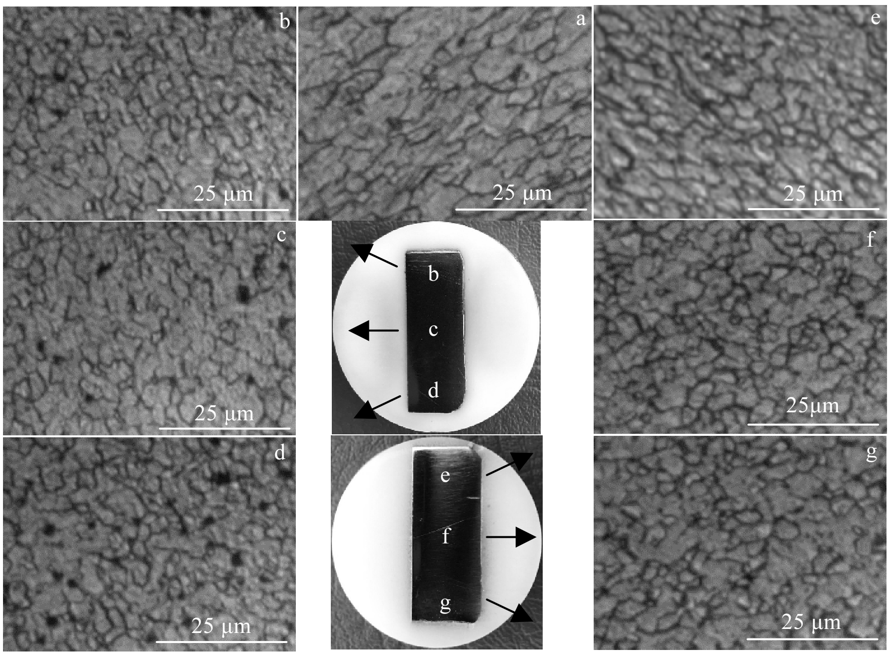

Fig.12 Microstructures of pure W before and after single pass ECDAP: (a) initial microstructure; (b~d) microstructure of primary shear zone; (e~g) secondary shear zone from inner to outer

zone and the secondary shear zone, respectively), it can be concluded that the degree of grain refinement was the largest in the inner angle zone.

Fig.13 shows the histogram of grain size distribution obtained from the secondary shear zone (about 2520 grains were measured). And the result reflects that the grain size is mainly between 1.0 and 3.0 μm with an average one of (2.0 ± 0.05) μm, approximately accounting for 92.97% of the counted grains. At the same time, the grains of the secondary shear zone were refined by 80% compared with the initial W, and the refinement efficiency increased by 27%~51% compared with the primary shear zone. Consequently, the grains of pure W can be refined to submicron-scale via ECDAP.

The grains will be elongated by shear force at ECAP angle. And the microstructure was refined and grains were elongated evidently after the first pass of ECAP; simultaneously, grain refinement did not appear to influence the electrochemical characteristics [12]. The grain elongation can be measured by axial ratio, which is the ratio of the long axis to the short axis of a grain. Fig. 14 shows the grain of Z plane after two shear deformations, in which obvious elongation of grains along shearing direction is observed. The aspect ratios of grains from

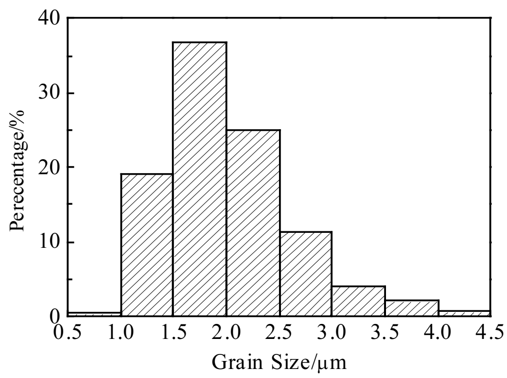

Fig.13 Grain size distribution in secondary shear zone of the ECDAP processed pure W sample

the inside to outside along the normal direction are 3, 1.3 and 2.3. It is revealed that the shear strain in the inner angle zone is severer than that in the outer angle zone, and the grain in the middle zone is practically equiaxed. Hence, the shear effect of double angle structure is superior. After ECDAP deformation, the sample presents the state of the middle equiaxed grain was wrapped up by elongated grain of outer layer.

3.5 Microhardness analysis

Fig. 15 illustrates the distribution of Vickers microhardness HV of the initial W and the X plane of the deformed sample. The microhardness of the initial W is approximate 4034 MPa, after ECDAP processing, it increased to 4830 and 4975 MPa in the primary and secondary shear zone, respectively. It can be explained by effective strain, viz., two shear deformation introduced severe shear strain, resulting in great grain refinement. Hence, the microhardness was enhanced because of the Hall-Petch strengthening [13] and strain strengthening. The microhardness of the secondary shear zone is 23% higher than the initial sample, and increase by 5% compared with the primary shear zone. Hao et al [14] expounded that the microhardness of pure W was improved by 8% after the traditional single pass ECAP (the inner angle is 120°and the outer angle is 30°) at 1073 and 1223 K. By comparison, the ECDAP process has great advantage on improving the mechanical property of pure W. In addition, Fig.15 indicates that the microhardness decrease at first and then increase from the inside to the outside along the normal direction in the shear deformation zone, which is in line with the distribution rules of the effective strain and grain size. Nevertheless, De et al [15] pointed out that the strain accumulation of the traditional ECAP deformation decreases gradually from the inside to the outside along the normal direction. Therefore, ECDAP can simultaneously improve the microhardness in the inner and outer angle zone, and make the microhardness of overall deformed sample more homogeneous.

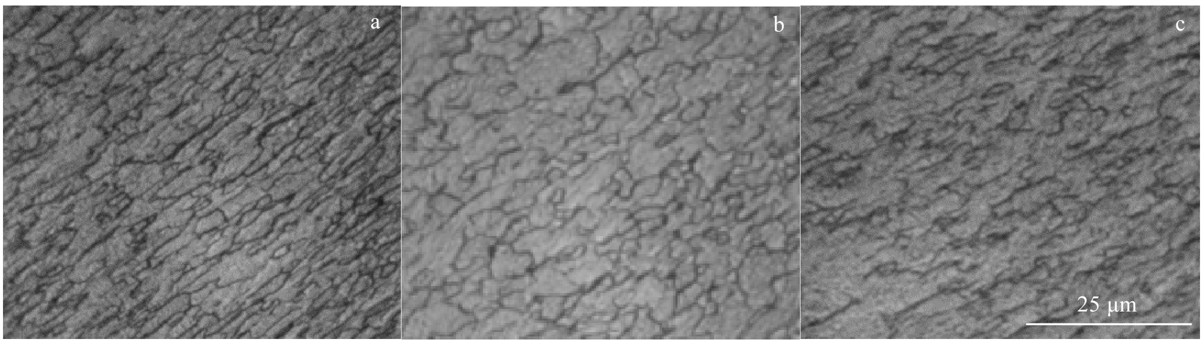

Fig.14 Grain distribution of secondary shear zone *Z* plane (a, b and c are the microstructures of secondary shear zone along the normal direction from inside to outside); grain aspect ratio: (a) 3, (b) 1.3, and (c) 2.3

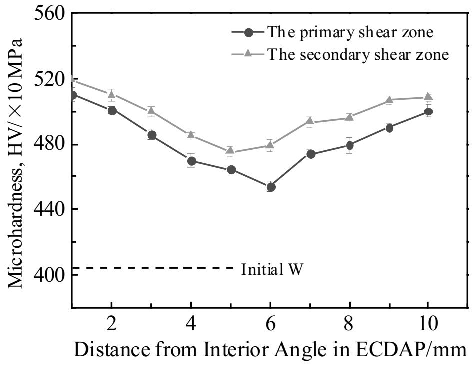

Fig.15 Microhardness distribution of pure W processed by single pass of ECDAP (the *I*-like error bar indicates the uncertainty of the measured data)

In order to evaluate the mechanical properties of pure W sample under single pass ECDAP, the average microhardness H_{ave} and the uniformity factor α were introduced. The H_{ave} was obtained by computing the microhardness values of the tested points in each deformation zone. The α can be calculated by the following Eq. (3).

$$\alpha = \frac{H_{max} - H_{min}}{H_{ave}} \quad (3)$$

where H_{max} and H_{min} are the maximum and minimum values of microhardness in each zone, respectively. Small α means that the distribution of microhardness is homogeneous [16]. The average microhardness and its uniformity coefficient in different zones are presented in Table 2. It is known that the uniformity coefficient of the secondary shear zone is smaller than that of the primary shear zone, so the distribution of microhardness in the secondary shear zone is more homogeneous.

Table 2 Average microhardness and uniformity coefficient of shear zone after single pass ECDAP deformation

Parameter	Primary shear zone	Secondary shear zone
H_{ave}/MPa	4830±60	4970±50
α	0.11	0.08

4 Conclusions

1) As a new SPD process, the ECDAP technology has been developed based on ECAP. Compared with ECAP, the local force of the die is improved, and the offset load of the punch is reduced. Meanwhile, when introducing equal strain, nevertheless, the uniformity of strain distribution is greatly improved by ECDAP.

2) The grain refinement of W can be achieved by the ECDAP method. The grain size of W decreases from 10 μm to 1.77 μm in shear deformation zone after ECDAP. In particular, the grains are refined enormously and the microstructure distribution becomes more uniform in the secondary shear zone. In addition, the grains are elongated obviously in the angle zone due to the severe shear deformation.

3) The microhardness (HV) of sample is improved from about 4034 MPa in initial state to about 4975 MPa after ECDAP experiment. The microhardness is more homogeneous in the secondary shear zone. Moreover, compared with traditional ECAP, ECDAP is more efficient to improve the microhardness of materials.

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纯钨等通道双转角挤压变形行为分析

李 萍，段自豪，吴 涛，华雅玲，薛克敏
（合肥工业大学，安徽 合肥 230009）

摘 要：作为一种新的大塑性变形（SPD）方法，基于等径角挤压（ECAP）开发了等通道双转角挤压（ECDAP）以减少冲头偏载。通过有限元模拟和实验研究分析了工业烧结纯钨在 1 道次 ECDAP 过程中的变形行为。结果表明：在相同工艺参数下，ECDAP 模具的局部应力分布更加均匀，冲头的偏载得到了改善。经 ECDAP 产生的平均等效应变与 ECAP 类似，但是试样经过 ECDAP 变形发生二次剪切变形，应变分布的均匀性大大提高。此外，在二次剪切变形区晶粒尺寸减小到 1.77 μm，并且在转角区晶粒被明显拉长。由于霍尔-帕奇（Hall-Petch）强化和应变强化作用使得显微硬度得到提高。以上结果表明 ECDAP 工艺是生产超细晶金属材料的一种有效方法。

关键词：纯钨；等通道双转角挤压；二次剪切；晶粒细化；显微硬度

作者简介：李 萍, 女，1973 年生，博士，教授，合肥工业大学材料科学与工程学院，安徽 合肥 230009, 电话: 0551-62901368, E-mail: li_ping@hfut.edu.cn