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ARTICLE

Mechanical and Tribological Properties of Commercially Pure Aluminum Processed by Equal Channel Angular Expansion Extrusion with Spherical Cavity

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Abstract: A novel severe plastic deformation (SPD) method, namely equal channel angular expansion extrusion with spherical cavity (ECAEE-SC), was introduced based on the modification of conventional equal channel angular extrusion (ECAE) process. By integrating expansion, shear and extrusion deformations in a single extrusion pass, ECAEE-SC process can induce larger accumulated strains into the billet, resulting in the significant grain refinement and improvement of the associated properties. In the present study, commercially pure aluminum (Al-1060) was subjected to one pass of ECAEE-SC process at room temperature, and two passes of ECAE process for comparison. The microstructure, Vickers hardness, tensile properties and wear properties of processed material were investigated. The results show that after one pass of ECAEE-SC process, the grains of ECAEE-SC processed aluminum are greatly refined with a typically elongated microstructure due to the high level strains induced in the material. The hardness and tensile strength of ECAEE-SC processed material increase significantly by almost 92.6% and 91.8% than those of the initial material, respectively. These improvements are considerately higher than those achieved by two passes of ECAE process. Moreover, the wear resistance of ECAEE-SC processed material is also enhanced. The worn surface morphology of ECAEE-SC processed sample presents the minimum width and depth of the wear scars, indicating that the wear mechanism is dominated by abrasive wear.

Key words: severe plastic deformation (SPD); mechanical properties; pure aluminum; tribological properties; wear mechanism

In recent years, ultra-fine grained (UFG) materials have received considerable attention in metals and alloys due to their well-developed microstructure and excellent properties[1]. As the most promising method for producing bulk UFG materials, severe plastic deformation (SPD) methods have been highlighted by many researchers and widely used to fabricate bulk materials in various application, such as aerospace and transportation industries[2,3]. Compared with other UFG fabrication techniques, SPD methods have outstanding grain refinement ability and simple process operation, which can be used to obtain bulk UFG materials with high density and purity in sub-micron or even nanometer level[4-6]. Among these methods, equal channel angular extrusion (ECAE) is the most representative and promising SPD method[7,8], which can produce shear strain on the material at the junction of channels without changing the cross-section of the processed billet. Therefore, high strains can accumulate by repeating the process through the desired

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passes, thus contributing to the UFG microstructure and the excellent mechanical properties. However, there are still some technical deficiencies in the practical application of conventional ECAE process[9,10], such as the restricted capability of single-pass grain refinement, the premature failure of the involved tools, the discontinuity of multi-pass extrusion, the poor uniformity of strain distribution, etc. Consequently, the development of new SPD method to increase the process efficiency is of great importance.

In the past decade, many researches were carried out to improve the abilities of conventional ECAE process. Moreover, some achievements have been made by combining traditional forming processes such as upsetting, forward extrusion and rolling with ECAE process. For example, Shen et al[11] fabricated pure magnesium (Mg) and Mg-0.6 yttrium (Y) binary alloy via casting followed by back-pressure assisted equal channel angular pressing (ECAP-BP). Xu et al[12] fabricated SiCp/AZ91 composites with uniformly distributed SiC particles and significantly refined the matrix grains by rotary-die equal channel angular pressing (RD-ECAP). Li et al[13] carried out a comprehensive investigation on Al-Zn-Mg-Cu alloy by equal channel angular dual-directional extrusion (ECADE), and a maximum strain of 3.97 with a large homogeneous deformation region was observed by SEM. El-Garaihy et al[14] investigated the influence of a novel SPD technique, multi-channel spiral twist extrusion (MCSTE), on the microstructural evolution and mechanical properties of AA1100 alloy, and it was found that the grain size of the as-processed billet significantly reduces by 72% after four passes of MCSTE compared with the grain size of initial material.

In the present study, a novel SPD process, namely equal channel angular expansion extrusion with spherical cavity (ECAEE-SC), was proposed to improve the capability of ECAE process. To obtain the comprehensive idea of ECAEE-SC process and evaluate its effect on the grain refinement and properties improvement of the processed material, a single-pass ECAEE-SC process was conducted on commercially pure aluminum (Al-1060) at room temperature. Then, the microstructure, hardness, tensile properties and the associated tribological properties of ECAEE-SC processed billet were investigated and compared with those of the material treated by two passes of conventional ECAE process.

1 Principle of ECAEE-SC

Fig.1 shows the schematic diagram of ECAEE-SC process. The designed die was modified based on the conventional ECAE process by integrating a spherical cavity and a horizontal expansion channel into the intersection of two equal cylindrical channels and the exit channel, respectively. During ECAEE-SC process, the moving punch pushed the billet downwards and gradually, and the materials expanded and filled the spherical cavity with increasing the diameter of the bottom part of as-processed billet. Then, this expanded part of the billet was subjected to shear deformation and extruded to its initial diameter. By continuing the process, more materials flowed into the expansion channel after a short rigid transformation. As all die containers were filled at the expansion stage during ECAEE-SC, a plug was placed at the end of exit channel for the first pass extrusion. After the filling of expansion channel, the plug was removed and the billet was extruded through the exit channel to its initial diameter, which was considered to be the back pressure for the expansion of the remaining material flow.

Moreover, it was worthy to mention that the spherical cavity and the expansion channel of the ECAEE-SC die, which caused an ideal hydrostatic stress state during the deformation, played an important role in the high strain accumulation and the considerable grain refinement of the processed material. After one pass of ECAEE-SC (1P-ECAEE-SC) process, the billet underwent various deformations containing expansion, shear and extrusion in a single die while the main dimensions remained the same, thus leading to the improvement of process efficiency.

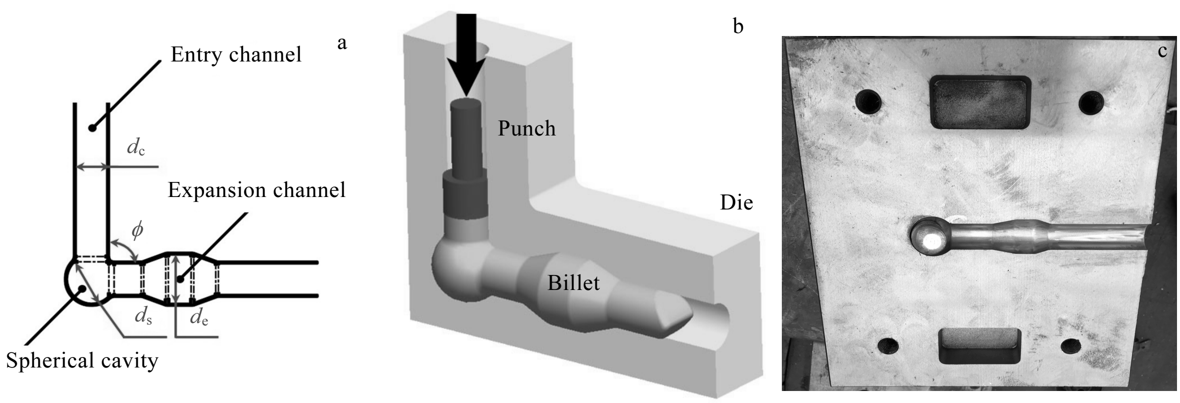

Fig.1 Schematic diagram of ECAEE-SC process: (a) die channel with geometrical parameters; (b) processed billet embedded in the ECAEE-SC die; (c) one half of the experimental ECAEE-SC die

2 Experiment

In the present study, commercially pure aluminum (Al-1060) with the purity more than 99.64% was selected as the starting material. Cylindrical billets with a diameter of 15 mm and a length of 80 mm were machined from the initial aluminum ingot. Before the ECSEE-SC deformation, all the billets were annealed at 150 ºC for 30 min followed by furnace cooling to homogenize the microstructure. In order to perform the ECAEE-SC process, a split die made from H13 tool steel with an inner angle of Φ=90°, the channel diameter of d_c=15 mm, the spherical cavity diameter of d_s=23 mm, and the expansion channel diameter of d_e=20 mm was constructed. A constant punch velocity of 1 mm/s was applied utilizing a hydraulic press for a single pass extrusion at room temperature. MoS_2 spray was used as a lubricant to reduce the interfacial friction between the die walls and the billet during the process. Moreover, to evaluate the efficiency of this novel ECAEE-SC process, two passes of conventional ECAE process via route A (the billet is repetitively pressed without any rotation) were also used under the same deformation condition for comparison.

After SPD operations, several samples were cut from the processed billets along the extrusion direction by a wire electrical discharge machining (WEDM). Then the microstructure of the cross-section of the processed samples after carefully grinding, mechanical polishing and electro-polishing, was observed by electron back scattering diffraction (EBSD). Microhardness tests were performed on the cross-section of the processed samples by KB30S microhardness tester with a Vickers indenter under a load of 100 g and a dwell time of 15 s. The measurements were carried out at 15 different selected points along the radial direction with equal spacing of 1 mm, and the average values were reported. The tensile test samples were machined from the central regions of the processed billets with gage sections lying parallel to the extrusion direction. The uniaxial tensile testing was conducted using a Shimadzu electronic universal testing machine of 100 kN with a constant strain rate of 0.01 s^{-1} at room temperature.

Wear tests were performed under dry sliding condition using a ball-on-disk tribometer (UMT-2) at room temperature. Prior to the wear test, samples were ground to $R_a \approx$ 0.16 μm with SiC papers (800# and 1000#), and then cleaned using anhydrous ethanol solution by ultrasonicator for 10 min, and finally dried. During the test, GCr15 steel balls were rubbed back and forth on the surface of the aluminum samples at a sliding speed of 2 mm/s. All samples were subjected to a load of 20 N for 20 min. A digital electronic balance was used to calculate the mass loss by measuring the initial and final mass of the samples. The wear resistance was characterized by the friction coefficient curves and the wear rate was calculated by Eq.(1)[15].

$$\text{Wear rate}=\text{Volume loss}/\text{Traveled distance} \tag{1}$$

In addition, to explore the wear mechanism of the processed samples during ECAEE-SC process, Inspect S50 scanning electron microscope (SEM) and Bruker Contour GT-K no-contact profilometer were employed to observe the three-dimensional micro-morphology of wear scars.

3 Results and Discussion

3.1 Microstructure analysis

Fig.2 shows the grain maps of the annealed and processed aluminum samples obtained by EBSD. As shown in Fig.2a, the annealed pure aluminum presents relatively homogeneous and coarse microstructure. The initial grains are nearly equiaxed with an average size of 400 μm. It is shown that two passes of conventional ECAE process lead to the significant distortion of the initial grains. The large equiaxed grains are elongated along the shear direction and the predominant slip system is visible in these elongated grains (Fig.2b). Therefore, the microstructure is considerably refined but with a high degree of inhomogeneity.

Moreover, due to the coupling effect of multiple deformations including upsetting, shear and extrusion during ECAEE-SC process, the deformation is essentially a simple shear deformation. Consequently, larger strains are induced into the processed material after just one pass of ECAEE-SC process, resulting in the more intensive shear-

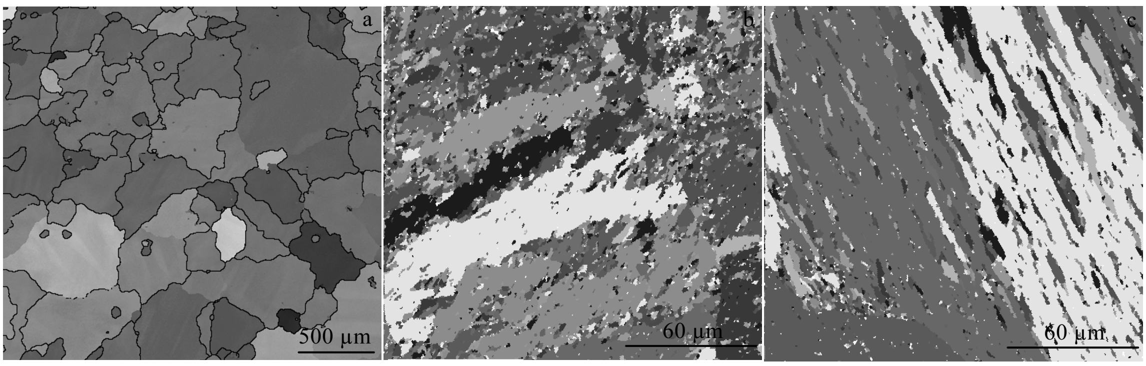

Fig.2 Grain maps of the annealed (a), two passes of ECAE processed (b) and 1P-ECAEE-SC processed (c) aluminum

ing and significant refinement. As shown in Fig.2c, there are a large number of narrow and elongated grains and several broken fine sub-grains in the microstructure. Comparing Fig.2c with Fig.2b, it is apparent that the grain refinement efficiency of ECAEE-SC process is considerably higher than that achieved by two passes of ECAE process. As a result, the shear strain plays an essential role in the grain refinement and this novel ECAEE-SC process has more excellent grain refinement ability.

3.2 Microhardness

Fig.3 represents the value of microhardness recorded along the radial direction on the cross-section of the processed samples in comparison with the samples under annealed condition. As shown in Fig.3, compared with the hardness of the annealed material, the microhardness of the 1P-ECAEE-SC processed aluminum increases from 366 MPa to 705 MPa with an acceptable homogeneous distribution, showing a significant improvement by more than 92.6%. This may be related to the dislocation accumulation generated by the intensive shearing under the high compressive stress state, causing the processed aluminum to exhibit strain hardening effect during ECAEE-SC process[16-18]. It is also clearly revealed that this considerable improvement is comparable with the case of ECAE processed samples, where the microhardness of the aluminum subjected to the 1st and 2nd pass of ECAE (1P-ECAE and 2P-ECAE) process reaching about 528 and 663 MPa, respectively. Moreover, the homogeneity of hardness distribution after ECAEE-SC process also improves. This observation suggests that the expansion deformation provided by the ECAEE-SC die is much more obvious than the improvement of mechanical properties for conventional ECAE processed material at extrusion stage. These observations of hardness behavior are in agreement with those reported by other researchers for multi-directional forging (MDF) processed pure nickel[1], and ECAP processed 6061 aluminum alloy[15].

3.3 Tensile behavior

Fig.4 illustrates the true stress-strain curves of the annealed aluminum as well as the processed samples. In addition, the corresponding parameters of tensile properties, including ultimate tensile strength σ_{UTS} and elongation to failure δ_f, are presented in Table 1. Based on the results shown in Fig.4 and Table 1, it can be seen that the annealed aluminum shows significantly lower strength but higher elongation. The tensile strength of ECAE processed aluminum increases from 98 MPa to 118 MPa after the 1st pass of extrusion, whereas the elongation decreases from 24.0% to 9.5%. As the number of passes increases, the tensile strength further increases to 173 MPa, although its elongation slightly decreases to 6.0%. Due to the possibly large imposed shear strain and obvious increase in dislocation density during ECAEE-SC process[2,3,15], a significant increase in the ultimate tensile strength of the aluminum is observed. After just one pass of ECAEE-SC process, the tensile strength reaches 188 MPa, increasing by 91.8% compared with that of the annealed aluminum. This improvement is considerably higher than that achieved by conventional ECAE process. However, the elongation reduces a little to 5.9%. This is attributed to the key feature of the expansion stage in ECAEE-SC die, in which higher accumulated strains and larger hydrostatic pressure are introduced to the processed material, thus improving the material plasticity and enhancing the mechanical properties.

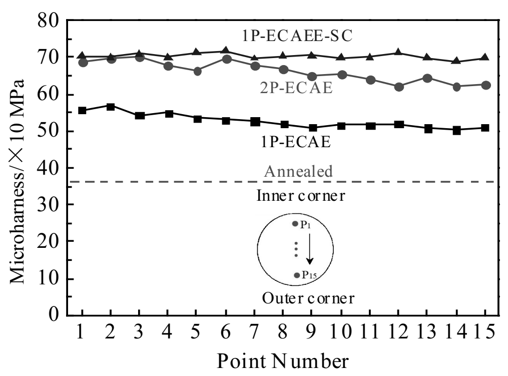

Fig.3 Microhardness at different sites of the processed and annealed aluminum

3.4 Wear properties

Fig.5 shows the average wear rate of the processed and annealed aluminum obtained from the wear tests. It is clearly observed that due to the intense plastic deformation imposed by SPD process, the wear rate of aluminum

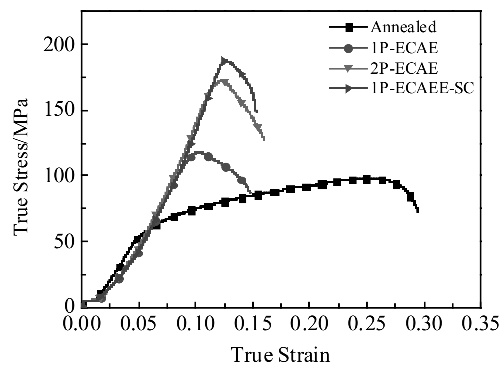

Fig.4 True stress-strain curves of the processed and annealed aluminum

Table 1 Tensile strength and elongation of the aluminum under different conditions

Condition	Tensile strength/MPa	Elongation/%
Annealed	98	24.0
1P-ECAE	118	9.5
2P-ECAE	173	6.0
1P-ECAEE-SC	188	5.9

significantly decreases. For instance, compared with the wear rate of annealed samples, the wear rate of samples decreases by 35.3% and 39.7% after the 1st and 2nd pass of ECAE, respectively. Furthermore, the wear rate of ECAEE-SC processed sample is lower than that of the 2nd pass of ECAE processed one, showing a reduction of wear rate of more than 31.4%.

Variations of the friction coefficient versus sliding time at 20 N load for the processed aluminum are shown in Fig.6. In addition, the friction coefficient is calculated by Eq.(2):

$$F=\mu N \quad (2)$$

where F, μ and N represent the frictional force, friction coefficient and load, respectively.

As shown in Fig.6, the trends of variation of friction coefficient curve in all cases are quite similar, which dramatically increases at the beginning, and then reaches a steady-state level. The average friction coefficient of annealed aluminum is around 0.651. Compared with the friction coefficient of sample under annealed condition, the friction coefficient reduces to 0.613, 0.574 and 0.526 after 1P-ECAE, 2P-ECAE and 1P-ECAEE-SC process, respectively. It is worth mentioning that the average friction coefficient of the ECAEE-SC processed material is much lower than that of the 2nd pass of ECAE processed material, indicating that this novel ECAEE-SC process leads to a better wear resistance performance. This claim is in consistent with the hardness behavior mentioned above (Fig.3). Significant reduction in the friction coefficient can be explained by the high efficiency of ECAEE-SC process, resulting in the induction of larger strains and significant improvement of mechanical properties of the processed aluminum[15,19].

3.5 Wear morphology

To investigate the wear mechanism of the processed aluminum during SPD process, surface morphologies of wear samples are shown in Fig.7. Base on the results, it is clear that the surface of the annealed sample is severely worn (Fig.7a). In addition to the adhesive wear debris and micro-ploughing grooves, the scale-like surface layer and spalling pits are observed along the sliding direction, implying that two wear mechanisms containing the spalling of oxide skin and slight abrasive wear are dominated. By continuing the process, the processed material experiences severe plastic deformation and large strains, resulting in a gradual decrease of the scale-like surface layer. Accordingly, the surface of worn samples becomes smooth and the surface roughness obviously decreases (Fig.7b and 7c). After one pass of ECAEE-SC process, the micro-ploughing grooves are parallel to each other and tend to distribute uniformly, and the depth and width further reduce, as shown in Fig.7d. This observation infers that by introducing high plastic strain into the material, the dominated wear mechanism of processed aluminum gradually changes to abrasive wear mechanism.

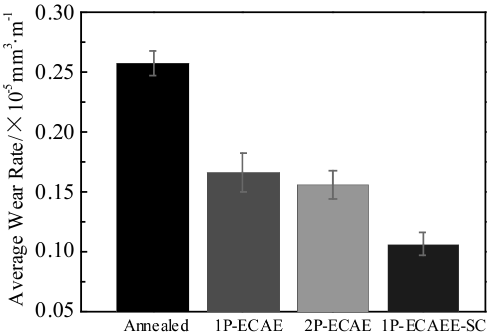

Fig.5 Average wear rate of specimens before and after different SPD processes

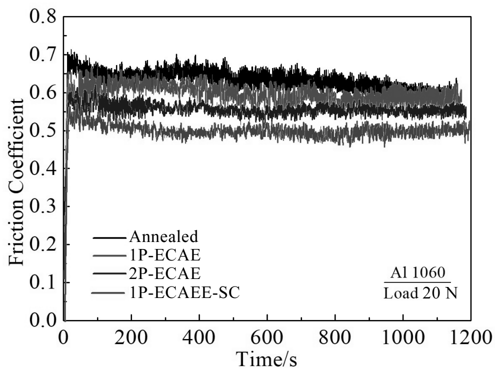

Fig.6 Friction coefficient curves of the aluminum under different conditions

Fig.8 shows the three-dimensional morphologies of wear scars under different conditions, in which the color scale on the right represents the depth of wear scar (Z direction). Furthermore, quantitative analysis is provided to explore the tribological behavior of aluminum during the SPD processes. For the annealed aluminum, the depth and width of wear scars are larger than those of samples subjected to SPD processes. With the increase of strain accumulation during SPD processes, the depth and width of the wear scars gradually decrease (Fig.8b and 8c). As the amount of plastic deformation strongly affects the wear resistance of the material, the ECAEE-SC processed aluminum has the minimum depth and width of wear scar (Fig.8d), exhibiting the best wear resistance. Moreover, the morphology characteristics of wear scars are in consistent with the results of microhardness variation mentioned above (Fig.3). Therefore, the novel ECAEE-SC process is capable of effectively improving the wear resistance of the material.

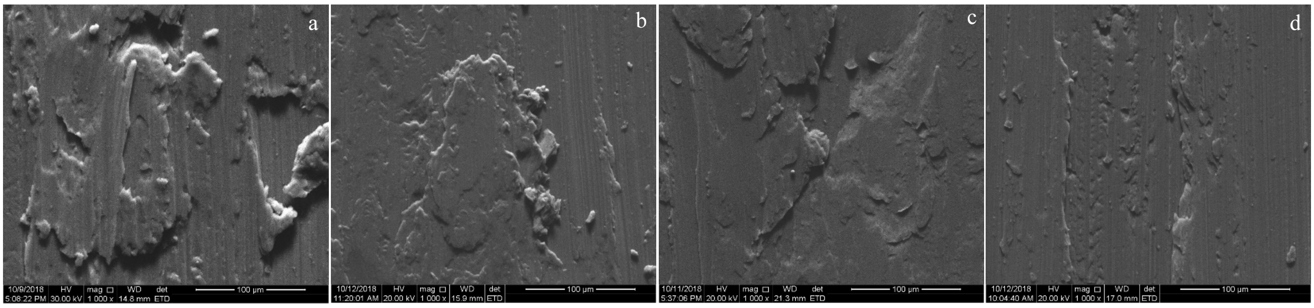

Fig.7 Wear surface morphologies of the aluminum under different deformation conditions: (a) annealed, (b) 1P-ECAE, (c) 2P-ECAE, and (d) 1P-ECAEE-SC

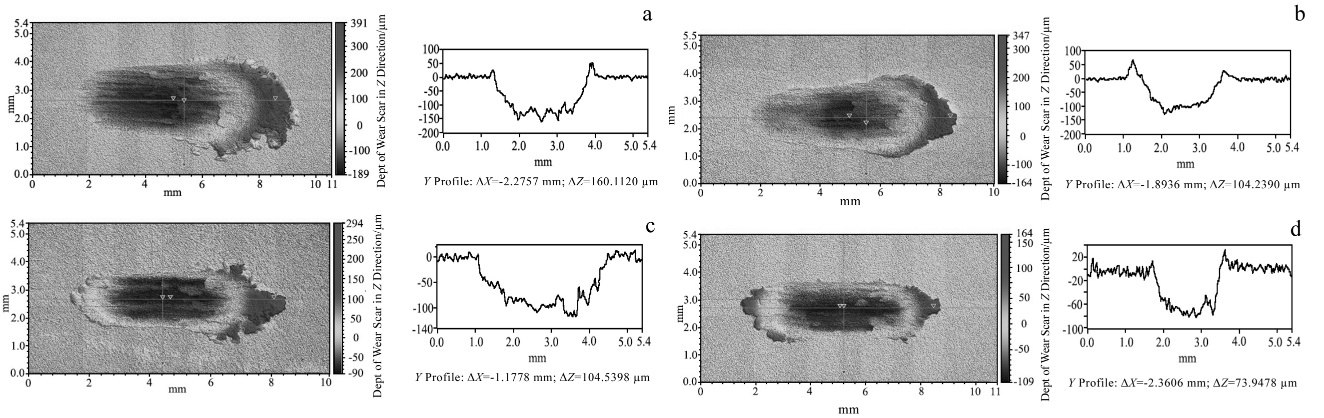

Fig.8 3D morphologies and 2D cross-sectional profiles of wear scars of the aluminum under different conditions: (a) annealed, (b) 1P-ECAE, (c) 2P-ECAE, and (d) 1P-ECAEE-SC

4 Conclusions

1) The novel equal channel angular expansion extrusion with spherical cavity (ECAEE-SC) process exhibits more excellent grain refinement ability than the conventional equal channel angular extrusion (ECAE) process. Because the intensive shearing, high level of strain and large hydrostatic pressure are generated in the ECAEE-SC processed material. Therefore, the microstructure of aluminum is severely broken into elongated and banded grains or even fine equiaxed grains after just one pass of the ECAEE-SC, which is more efficient than the 2 passes of ECAE process.

2) Due to the great grain refinement, the mechanical properties of processed aluminum improve significantly. After just one pass of the ECAEE-SC, the average microhardness increases from 366 MPa to 705 MPa with an acceptable homogeneity. Moreover, there is a notable increase in tensile strength from 98 MPa to 188 MPa. The strength improvement during ECAEE-SC process is comparable to that of sample subjected to 2 passes of the ECAE process, while the elongation of the processed sample decreases slightly.

3) The wear resistance of the aluminum significantly improves after a single pass of the ECAEE-SC. The wear surface morphology of processed aluminum exhibits the minimum width and depth of the wear scars, indicating that the wear mechanism changes from the oxidation and delamination wear under the annealed condition to the mixed wear mechanisms dominated by abrasive wear after ECAEE-SC process.

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工业纯铝等通道球形转角膨胀挤压变形的力学与摩擦学性能

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摘 要：对传统等通道转角挤压工艺（equal channel angular extrusion, ECAE）进行改进，提出一种新型剧烈塑性变形法（severe plastic deformation, SPD）—— 等通道球形转角膨胀挤压（equal channel angular expansion extrusion with spherical cavity, ECAEE-SC）。该工艺通过耦合镦-剪-挤等多种变形效应，可在单道次挤压下实现坯料内部较大的塑性应变累积，进而获得理想的晶粒细化与性能提升效果。在室温条件下采用 ECAEE-SC 工艺对工业纯铝（Al-1060）进行单道次挤压，并与相同条件下的 2 道次 ECAE 处理变形结果进行对比。采用 EBSD、SEM 等测试手段，研究了工业纯铝经 ECAEE-SC 变形晶粒特征与磨损表面形貌，并测试了变形材料显微硬度、拉伸性能与摩擦学性能。结果表明，在 ECAEE-SC 工艺剧烈塑性应变诱导下，工业纯铝经单道次挤压变形后晶粒显著细化，呈典型的剪切条带状特征。与初始退火态相比，变形材料显微硬度与抗拉伸强度分别提升了 92.6%和 91.8%，且性能提升效果明显优于 2 道次 ECAE 变形。同时，ECAEE-SC 工艺有效提高了工业纯铝的耐磨性能，工业纯铝变形后表面磨痕宽度最小，磨痕深度最浅，其磨损机理以磨粒磨损为主导。

关键词：剧烈塑性变形；力学性能；纯铝；摩擦学性能；磨损机理

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