

The effects of microstructure and its properties on the annealing treatment for ultra-fine grain of 1060 commercial pure aluminums processed by accumulative roll bonding (ARB)

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Abstract: In this study, ultra-fine grain of 1060 commercial pure aluminums was processed by accumulated rolling bonding (ARB) technology. Effect of microstructure and mechanical properties on the annealing treatment for ultra-fine grained was investigated. The results reveal that, 1060 commercial pure aluminums which was processed after 8 passes ARB at room temperature, grain size of non-deformed was fined from 41 μm to 0.42 μm , the strength increased 2.5 times, but the elongation ratio decreases. The SEM photos show that the material plastic property drops sharply after being processed, but it is gliding fracture. The samples of ARB different passes receive annealed treatment for 1h at 150 $^{\circ}\text{C}$, because annealing treatment improve the interfacial bond strength of compositing materials, the strength increase a little. The samples after ARB5 passes were annealed for 1h below 200 $^{\circ}\text{C}$, microstructure was in recovery level. Grain size within 0.47 μm ~ 0.58 μm , and mechanical properties were stabilization. Above 200 $^{\circ}\text{C}$, grains were recrystallized and the grain size grows up, mechanical properties of materials recovered.

Keywords: Accumulative roll bonding; ultra-fine grain materials; 1060 commercial pure aluminums; annealing

1. Introduce

Grain refining can increase material strength and toughness to improve materials properties at ambient temperature. Severe Plastic Deformation (SPD) is one of the effective ways to make bulk materials with Ultra-fine grains. Up to the present, much research has been done on pure Aluminum & Aluminum alloy, IF Iron, and Copper which have been processed by severe plastic deformation, special processing technique such as equal channel angle press (ECAP)[1,2], high pressing torsion (HPT)[3], multi-directional forging (MF)[4], cyclic extrusion compression (CEC)[5], accumulative roll bonding (ARB) [6,7], etc. These materials achieve higher plastic strain, effectively make the coarse grains fine and increase the properties of materials. However, after severe plastic deformation, the stored energy inside the smaller grains is higher and it begins to grow bigger at low temperature. Therefore, the research on structure stability of ultra-fine grain material is focused on to improve the stability of the ultra-fine grain and to develop and take good advantage of the properties of the ultra-fine

grain materials.

ARB created by Japanese scholar Saito et al. is a method of severe plastic deformation to produce bulk sheet with ultra-fine grain structure in large quantity. Outstanding characteristic of ARB is made through many times repeated roll bonding. Materials made in this way have large accumulated strain. And the size of sheet is not changed after rolling so that the structure inside is refined to improve the properties. However, there are few reports on the influence on the microstructure and physical properties of the bulk ultra-fine grain material produced by SPD through annealing treatment. Because deformation stored energy is very high after the severe plastic deformation in materials, at certain temperature, the grain inside is easier to grow up compared with the common materials and it has an important influence on the microstructure and mechanical properties. Therefore, more and special research on the stability of the microstructure and mechanical properties of the ultra-fine grain materials upon annealing of this type of materials should be done in the future.

In this paper, the author attempts to make a study on the influence of microstructure and mechanical properties of 1060 industrial pure aluminum produced by ARB technique, as well as effect of ultra-fine grain materials upon heat treatment.

2. Experimental

In order to study the influence of microstructure of materials after ARB process and effect of ultra-fine grain materials upon heat treatment, 1060 commercial pure aluminum is chosen. Its chemical component (mass fraction) is: 0.0025Si, 0.0035Fe, 0.0005Cu, 0.0005Zn, the rest-Al. The material is treated by fully annealing at 400°C for 1h before the ARB process. The initial dimensions of the material are 1.84mm in thickness, 100mm in width and 300mm in length. The steel wire brush and acetone is used to clean the surface of the combining surface of the 2 samples and the oxidation layers and oils. Furthermore, it will make the surface of the sample with a certain roughness. And then the two piece samples are made to stack, and rolled by rolling machine without lubricating and no less than 50% reduction each pass at room temperature. The sheet after rolling is cut into 2 pieces and surface treatment again. Rolled and compound again and again. Materials have been made 8 cycles processed of ARB for the test.

The rolling is done on a two-high mill which roll diameter is 170mm at room temperature. The roller peripheral speed is 0.42m/s, so that the strain rate is 23/s.

The annealing test on different ARB passed samples are done in a Box-type resistance furnace at 150°C for 1h. Choose the sample of good interface after ARB 5 passed (Equivalent strain is 4.0) at a temperature of 100°C, 150°C, 200°C, 250°C, 300°C, 350°C, 400°C, for 1h to test what the influence will be on the microstructure of ultra-fine commercial pure aluminum made by ARB at different annealing treatment temperature. The samples are air cooled after annealing.

The observation of the microstructure of the base material and the ARBed samples with different passes rolled and under various annealed conditions are made with the JEM-2—CXTEM transmission electron microscope, which accelerates voltage of 120kV. The thin foil samples are prepared by twin-jet electro-polishing using a 200ml HNO₃ and 400ml CH₃OH solution. Tensile strength test at room temperature are carried out using a INSTRON—

1341 elongation apparatus, samples' quantity no less than 3 pieces of each passes, loading rate: 1.0 mm / min, and maximum load: 10KN. Observe the fracture of every tensile sample with Quanta 200. Test the hardness of the surface with micro hardness tester, loading: 100gf, time: 10s.

3. Results and Analysis

3.1 The Changes of materials after ARB at room temperature.

3.1.1 Microstructure

Fig.1 shows the TEM microstructure of 1060 commercial pure aluminum after various passes of ARB. The microstructure of the original samples shows on Fig.1a, and the mean grain size before ARB processed was about 41 μ m. After ARB1 passed at the room temperature, the materials is mainly composed of dislocation cells and sub grains (Fig.1b). The density of the dislocation inside the grain increases to make a big quantity of dislocation cells. There are millions of dislocation cells on the cell wall and also the dislocation inside of the grain is less than on the wall. The microstructure after ARB2 and ARB1 is more or less the same. But with the increase of the rolling passes, the size of the dislocation cells correspondingly reduces and the dislocation density is much thicker on the wall after ARB3 passed. The energy of the material increases after the mass deformation which causes the cross slip and dynamic recovery. The microstructure can turn sub-grains by the process of dynamic recovery while the wall of the cells becomes the sub-grain boundary (Fig.1c). The microstructure after ARB 5 to ARB 8 passed is similar (Fig.1d.1e.1f), and the dislocation cell shaped microstructure is unique and the shape of the grains is very clear. The ultra-fine grain takes up a bigger portion with the increase of the rolling passes, but the size of the grains is not the same. The average size of the grains is about 0.42 μ m after ARB 8 passed, surrounded by clear grain boundaries. There exists oriented angle gap and also some sub-grains are left.

Fig.2 is the TEM of the sample after ARB at 150 $^{\circ}$ C annealing for 1 hour. The comparison between Fig.1b, 1c, 1d and 1e shows that the lath shaped grain microstructure becomes equiaxed grains, but the misorientation of the formal deformation microstructure still exists. The dislocation cell inside reduces but the sub-grains increases. The density of the dislocation is much less than unannealed. But the grain size doesn't change a lot which means it's on the stage of recovery, hence showing good stability.

3.1.2 Mechanical properties

Fig.3 shows that the mechanical properties of the materials are improved after ARB processed at room temperature. The increase of the Tensile strength after ARB 1 is the fastest, from 87MPa before ARB to 149.5MPa, totally 62.5MPa added. With the increasing of the rolling passes, the tensile strength improves as well, and after ARB 7 passed, it reaches the maximum as 217.5MPa, 2.5 times than the original.

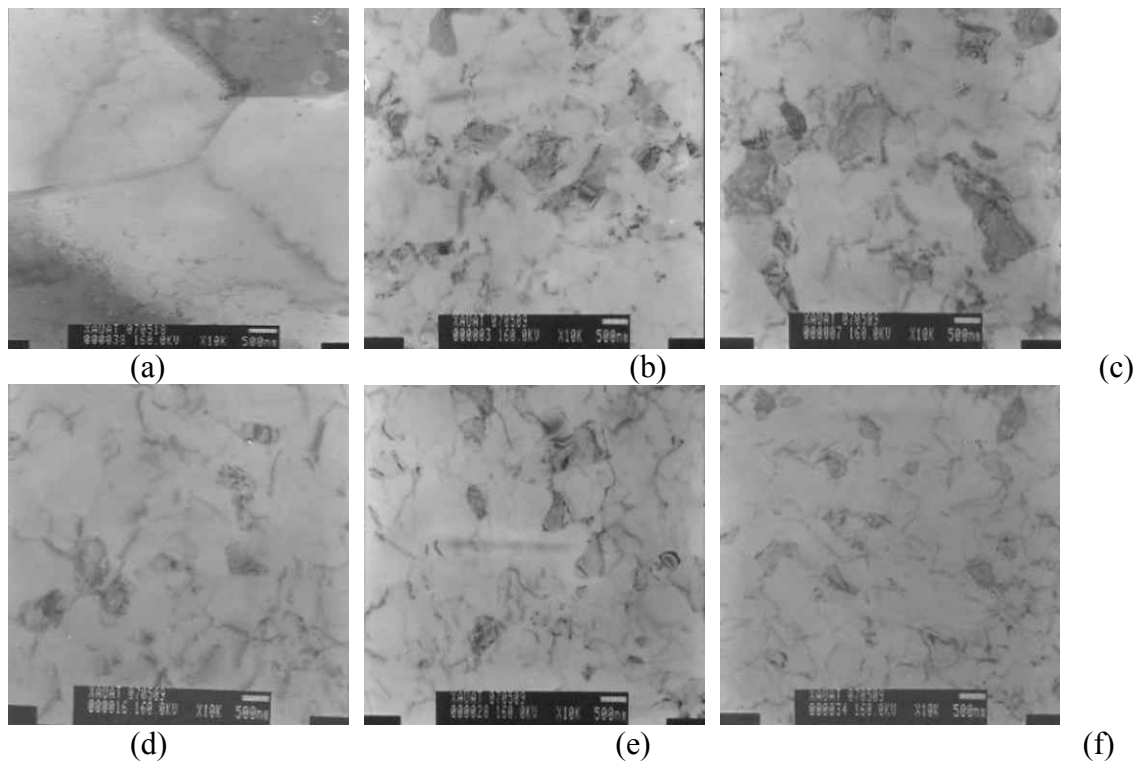


Fig.1 TEM micrographs showing the microstructure change of 1060 commercial pure aluminums after ARB processed: (a)Before rolling; (b)ARB1-passed; (c)ARB3-passed; (d)ARB5-passed; (e)ARB7=passed; (f)ARB8-passed

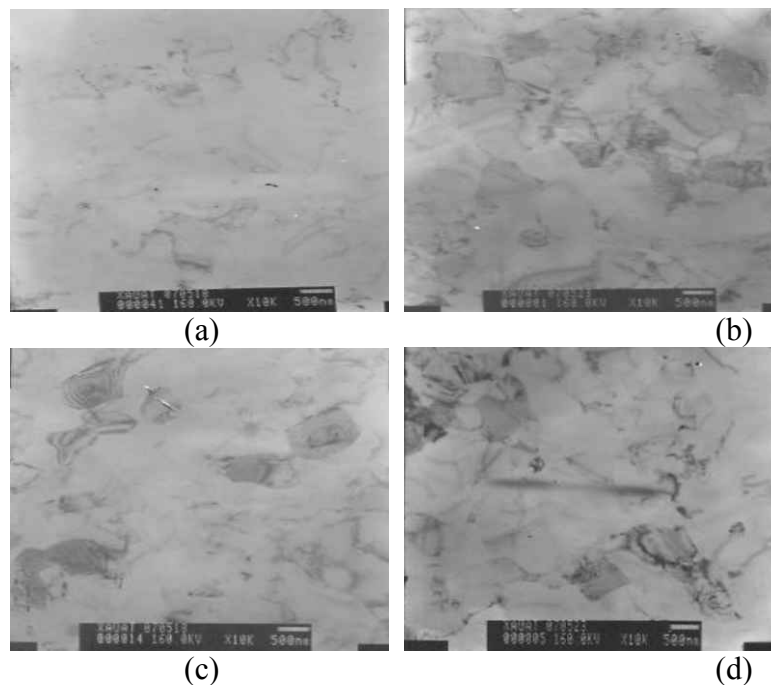


Fig.2 TEM micrographs showing the microstructures of 1060 commercial pure aluminums after ARB processed annealing at 150°C×1h: (a)ARB1-passes; (b) ARB3-passed; (c)ARB5-passed; (d)ARB7-passed

But meanwhile, the elongation rate drops a lot after ARB 1, from 46% before ARB to 6.42%. With the increase of deformation, it has no big influence and it's 7% more or less

(Fig3b). It shows that there exists more dislocation inside and the density of dislocation is thicker during the ARB severe deformation processing. Because of the mutual interactions between each other, the internal stress and work hardening also appears. The tensile is increasing while the ductility reducing. With the increase of ARB passes, the quantity of smaller fine grains is increasing and after ARB 5 passed, the ultra-fine grains are distributed homogeneously inside, which is the key reason that the tensile strength and elongation rate changes slowly.

After annealing treatment in 150 for an hour, it has a little increase on the tensile strength, because it's on the stage of recovery and the defects inside of the grains are gone and the internal stress are released. Dislocation inside is rearranged and forms some grain boundaries with small angle and also moves to the boundary. And meanwhile, the annealing treatment also helps improve the bonding strength so as to increase the tensile strength a little. But there is no big difference on elongation rate before or after the treatment.

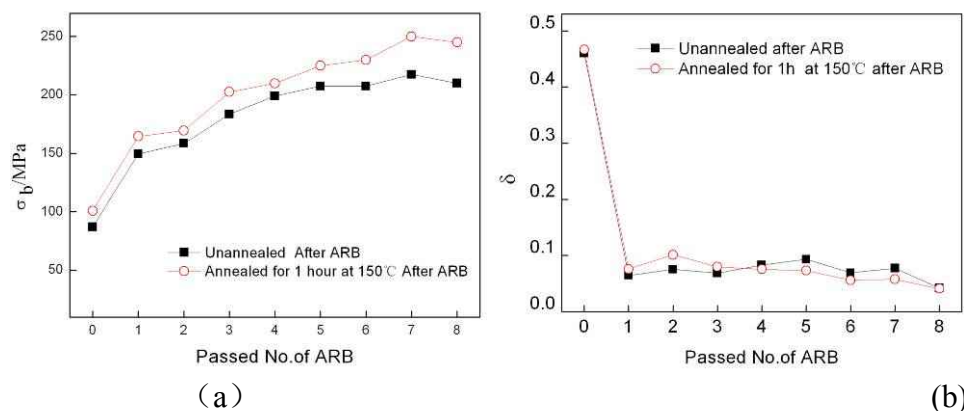


Fig.3 σ_b , δ Versus No. of ARB after unannealed and annealed: (a) σ_b , (b) δ

3.2 The changes of the microstructure and properties after annealing

3.2.1 Microstructure

Fig.4 shows the TEM microstructure of the samples after ARB 5 passed annealing at temperature from 100°C to 400°C for 1 hour. From Fig.4a to 4c, there is no clearly change between the microstructure of the sample at 200°C and the rolled at room temperature after ARB 5 (Fig.2c). There are some unclear boundaries or extinction fringes, which shows the microstructure is on the stage of recovery and the grain size doesn't change more. The dislocation in sub-grains moves to the sub-grain boundaries, the sub-grain and ultra-grain begins to grow. The grain size grows from 0.47 μ m to 0.58 μ m slowly. After annealing at 250°C, the dislocation cells reduce, and the grain boundary becomes clearer. The recrystallization grains, small unrecrystallization grains and sub-grains exist together, and the dislocation density in recrystallization grains is less than that in recrystallization grains (Fig.4d). Annealing at a temperature over 300°C, the grain boundary is clear and grains grows apparently. And there exists some dislocation (Fig.4e). With the increase of temperature, recrystallization microstructure mainly exists inside the material. After annealing at 400°C, the average size of the grains is over 20 μ m (Fig.4f). It shows that the microstructure of ultra fine grain of 1060 commercial pure aluminum has some stability below the temperature of 200°C.

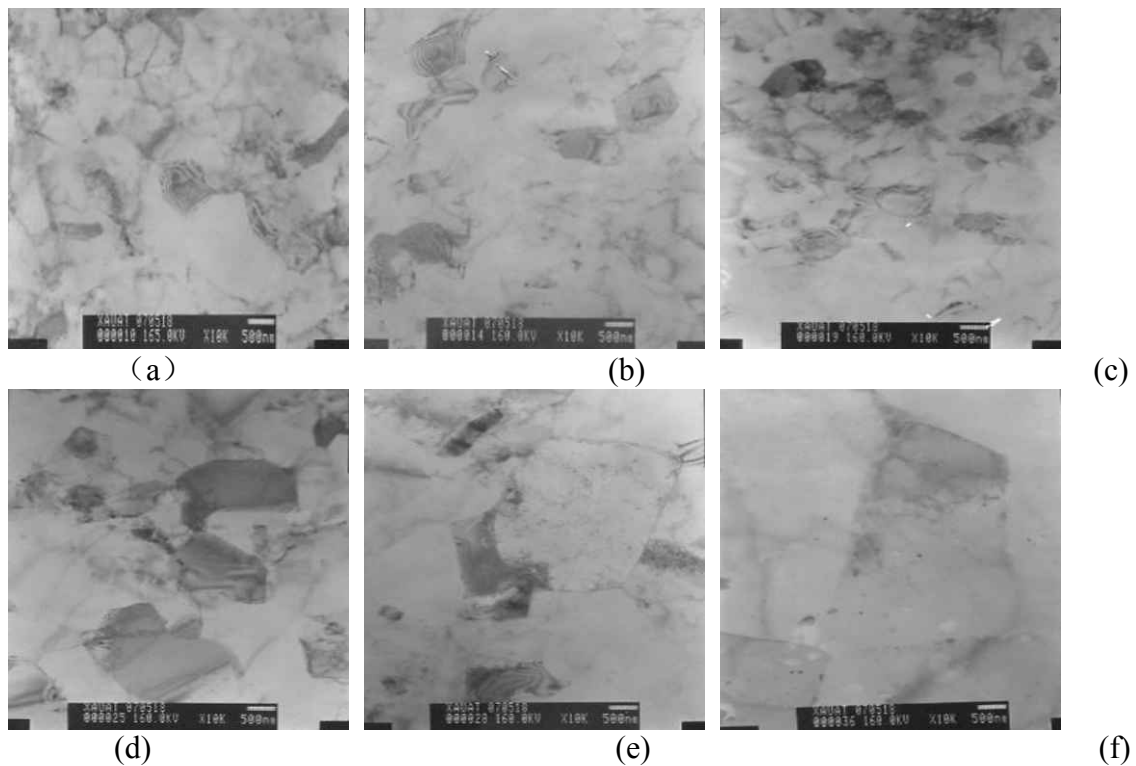


Fig.4 TEM micrographs showing the microstructures of 1060 commercial pure aluminums after ARB 5 processed annealing for 1h at 100°C ~400°C: (a)annealed at 100°C; (b)at 150°C; (c)at 200°C; (d)at 250°C;(e)at 300°C;(f) at 400°C

3.2.2 Mechanical properties

Fig.5 shows the mechanical properties of the materials after ARB 5 passed annealing at temperature from 100°C to 400°C for 1 hour. The tensile strength of the material can be divided into 3 parts in Fig.5: 1. Below 150°C, the tensile strength is stable, basically 220MPa; 2. Between 150°C ~300°C, it reduces rapidly from 222MPa to 111MPa; 3. Over 300°C, it has a little change on its tensile strength. At 400°C, it reduces to 99MPa. The change of the elongation is corresponding to the tensile strength. Below 200°C, there is a slight change on elongation, basically 6%. Between 200°C ~350°C, the elongation increases quickly, from 6% to 36%, and then it becomes flat. It shows that the ultra fine grain of 1060 commercial pure aluminum after ARB 5 passed is on the stage of recovery and the microstructure in the matrix doesn't change a lot and the internal stress reduces and its mechanical properties changes a little slightly after annealed below 200°C. For annealing over 200°C, the recrystallization grains happen, and its grains grows faster and the quantity increases. And the microstructure becomes recrystallization microstructure from the deformation microstructure. The tensile strength reduces while the ductility increases. With the increasing of the temperature, the grains grow consistently and the mechanical properties begin to recover. However, the microstructure of materials of ARB5 passed after annealing over 400°C doesn't reach the condition of the original materials, so the tensile and elongation can't reach the original data.

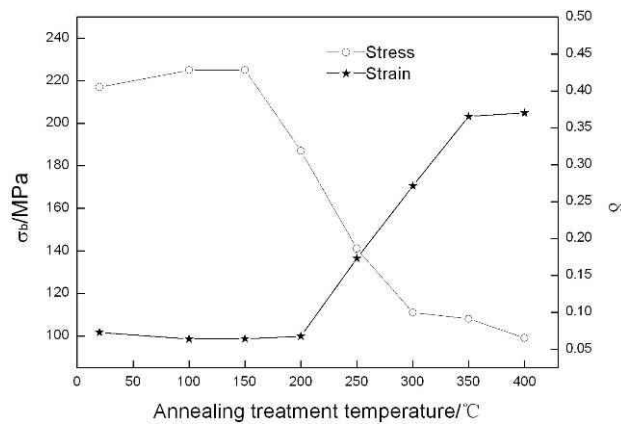


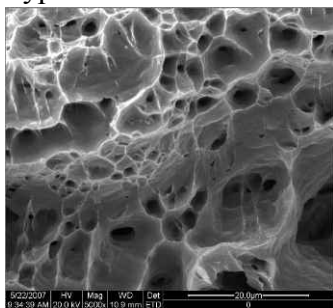
Fig.5 σ_b , δ Versus No. of ARB5 after annealed at different temperature

3.3 Analysis on the tensile fracture

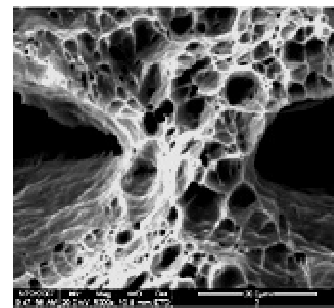
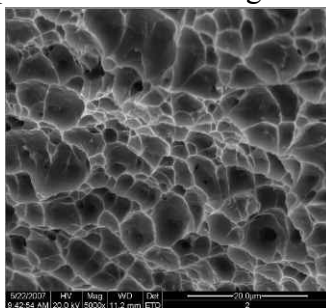
Fig.6 shows the scanning morphology of the tensile fracture after different ARB passes without annealing. The fracture of the base material is the typical fracture of ductile, existing big and deep dimples, and the non-uniform distribution of dimples (Fig.6a). After ARB1 passed, the dimple on the fracture becomes smaller and lighter, and it changed more uniform than before ARB processed (Fig.6b). After ARB3 passed, it's clearly seen that the matrix in some parts of the boundaries of the materials interpenetrates, which shows that it still exists some unclosed parts inside though the composition face combines closely (Fig.6c). The morphology of the fracture after ARB5 (Fig.6d) and ARB6 (Fig.6e) passed shows it doesn't change great, many of which are small and undep dimples homogeneously. After ARB 8 passed, it is shown that there are some holes after the tensility of material which influences on the ductility of the materials. .

This is why work hardening has a big influence on the ductility of the materials. And it shows that the extent of the grain fining is bigger because rolling deformation is distributed homogeneously inside the material from the morphology of the fracture after ARB 5 passed. With the increased of ARB passes, the grain size doesn't change during the rolling process, so its tensile property didn't change a lot and there was no obvious change on the sample's fracture.

The morphology of the tensile fracture after ARB processed shows they are all dimples inside, and there are some smaller dimples distributed inside the bigger dimples, which shows that the fracture happens on the tensile processing. There is plastic deformation during the process and under the tensile stress, the edge of the dimples breaks and penetrates mutually. So it's the typical fracture with microporous accumulating.



(a)



(b)

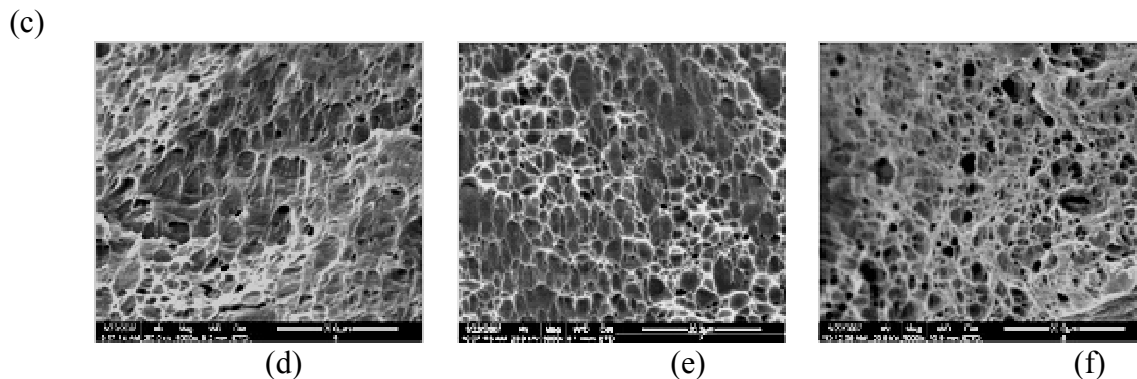


Fig.6 Tensile fracture morphology of 1060 commercial pure aluminums after ARB passed: (a)Before rolling; (b)ARB1-passed; (c)ARB3-passed; (d)ARB5-passed; (e)ARB7-passed; (f)ARB8-passed.

4. Conclusions

(1) 1060 commercial pure aluminum is rolled by 8 cycles of ARB passed at room temperature, the accumulated equivalent strain is 6.4, and the ultra fine grain of 1060 commercial pure aluminum is made. The grain size is fined from $41\mu\text{m}$ to $0.42\mu\text{m}$ after ARB8 passed and the tensile increases from 87MPa to 217.5MPa, 2.5 times of the original materials. The yield strength reduces from 46% to 6.42%, and with the increase deformation amount, it is more less 7%.

(2) Under the small strains, small dislocation cells are formed and with the increase of deformation amount, and the dislocation cells turn ultra-fine sub-grains. And under severe deformation, ultra fine grains are formed. The microstructure and properties of the sample materials have a good stability and the strength increases meantime after the samples is ARB differently passed and annealing at 150°C for an hour.

(3) After ARB 5 passed, the accumulated equivalent strain is 4.0. After annealing below 200°C for 1 hour, the grains grow slowly and the average size is about $0.58\mu\text{m}$.and the microstructure is on the stage of recovery, so the microstructure and the properties of ultra fine grain material is very stable.

(4) From the morphology of the tensile sample after ARB passed, the size of dimples becomes smaller and lighter with the increase of ARB passes. After ARB 8 passed, there are many microporous formed in the materials during the tensile testing, and the fracture pattern is ductile fracture.

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