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**Tailoring strength-ductility of Al_{0.8}CoCrFeNi/Al composites
fabricated by deformation-driven metallurgy via incorporating high-
energy ball milling pretreatment**

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Abstract

High-energy ball milling (HEBM) was utilized as the pre-treatment procedure to tailor the mechanical properties of high entropy alloy (HEA) reinforced aluminum matrix composites fabricated by deformation-driven metallurgy. The evolution of the milled powders as well as the fabricated composites was investigated via prolonging the holding time during HEBM. HEBM pretreatment is proved to be effective in refining the incorporated reinforcements and introducing massive dislocations, which were conducive to the mechanical properties of the fabricated composites. With prolonging the holding time of HEBM, the average size of reinforcements in the fabricated composites decreased and the ratio of recrystallized grains increased. Besides, the yield strength as well as the ultimate tensile strength of the fabricated composites increased while the elongation decreased. When the holding time of HEBM reached 25 hours, the yield strength, ultimate tensile strength and elongation of the fabricated

composites reached 282 MPa, 341 MPa and 12.4%, which proved that the pre-treatment of HEBM effectively improved the strengthening effect of HEAs reinforcements.

Keywords

Deformation-driven metallurgy; High-energy ball milling; High entropy alloys; Composites; Recrystallization; Mechanical properties

1. Introduction

Aluminum (Al) matrix composites (AMCs) with outstanding performances have been researched and developed rapidly under the urgent demand of energy-conservation and emission-reduction [1–3]. Particles of high entropy alloys (HEAs) have been proved to possess the great potentials as the ideal reinforcements for AMCs, due to the good interfacial wettability with Al matrix as well as their high strength, hardness, ductility, outstanding anti-corrosion properties and so on [4–6]. At present, AMCs reinforced by HEAs have been prepared by stirring casting [7,8], squeeze casting [9,10], vacuum suction casting [11], squeeze infiltration [12], laser powder bed fusion [13–15], selective laser melting [16], vacuum hot-pressing sintering [17,18], spark plasma sintering [19–21], microwave sintering [22,23], powder hot extrusion [24,25], friction deposition [26], friction stir processing [27,28], mechanical alloying [29,30] and so on. Overall, HEA/Al composites fabricated by the fabrication processes mentioned above still existed some problems such as serious interfacial reaction, non-uniform distribution of reinforcements and also the complex preparation processes [31]. Combining the advantages of powder-metallurgy process and friction stir processing, we proposed a solid-state fabrication process named deformation-driven metallurgy (DDM), by which the bulk AMCs could be directly fabricated from the as-received powders or powder precursors [32–36]. Under the effect of severe plastic deformation during DDM process, grain refinement of Al matrix, uniform dispersion of HEAs

reinforcements as well as the inhabitation of brittle intermetallic compounds at the HEA/Al interface are achieved, thus the existed problems mentioned above could be solved concurrently [37–39]. Based on this, HEAs reinforced AMCs with outstanding mechanical properties are obtained.

The refinement and uniform dispersion of HEAs reinforcement in Al matrix hinder the movement of dislocations, which could restrict the growth of recrystallized grains during DDM process and also enhance the interaction with dislocations during plastic deformation [40,41], which leading to the furtherly enhancement of mechanical properties. High-energy ball milling (HEBM) is widely utilized as the pretreatment procedure of powder metallurgy processes, during which the ball-milled powders are deformed, broken and refined under the effect of mixing, grinding and striking [42–44]. Besides the refinement and homogenization of reinforcement derived by HEBM, high-density dislocations are introduced into powders, which will promote the recrystallization process and thus enhance the mechanical properties of the fabricated bulks [45,46].

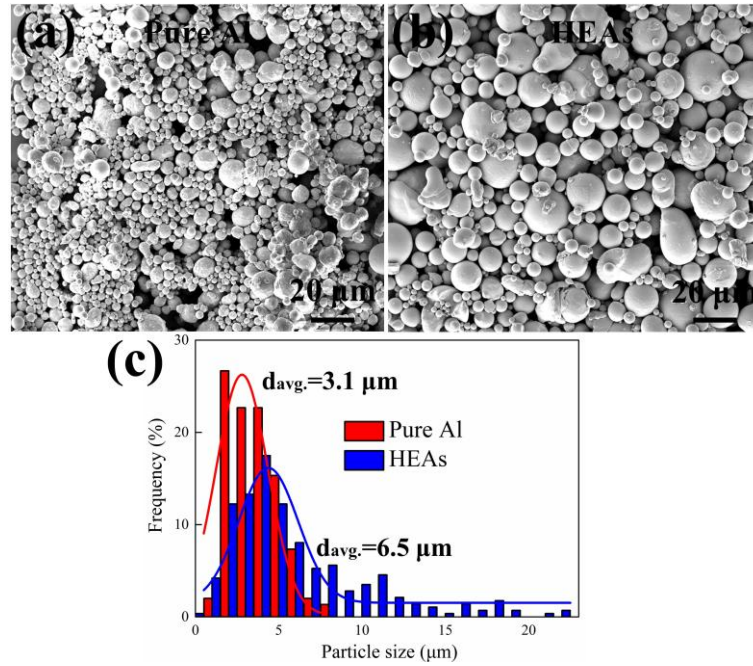
Here in this paper, mixed powders with Al and HEAs particles were pretreated by HEBM to refine HEAs reinforcements and also increase the dislocation density of Al particles. Meanwhile, the effect of HEBM duration times on the evolution of the mixed powder precursors were explored, including the powders' morphology, size and microstructures. Bulk AMCs were fabricated utilizing the HEBMed powders by DDM, and the microstructural evolution and mechanical properties of the fabricated AMCs were characterized, in order to regulate the strength-ductility of the fabricated AMCs.

2. Materials and experiments

2.1 Morphology and compositions of the as-received powders

Fig. 1 illustrates the morphology and size distributions of the as-received vacuum

1 gas-atomized pure Al particles ($\sim 10\ \mu\text{m}$) and HEAs particles ($\sim 25\ \mu\text{m}$), and Table 1
 2 exhibits the detailed chemical compositions of the utilized HEAs particles. The as-
 3 received powders are spherical and the average particle size of the as-received pure Al
 4 and HEAs are calculated to be $3.1\ \mu\text{m}$ and $6.5\ \mu\text{m}$, respectively. The nominal
 5 composition of the as-received HEAs particles is calculated to be $\text{Al}_{0.8}\text{CoCrFeNi}$, as
 6 exhibited in Table 1.



7
 8 **Fig. 1.** Morphology of (a) pure Al particles, (b) HEAs particles and (c) size distribution of the
 9 as-received powders.

10 **Table 1** Chemical compositions of the as-received HEA particles (at, %)

Element	Al	Co	Cr	Fe	Ni
At. %	16.80	20.74	20.49	21.27	20.70

11 2.2 HEBM pretreatment of the mixed Al-HEAs powders

12 HEBM was conducted by planetary ball-milling machine (QM-3SP2, Nanjing
 13 Nanda Instrument Equipment Co., Ltd) with vacuum ball-milling tank (stainless-steel,
 14 250 mL in volume) and grinding balls (ZrO_2 , 300 g in weight). The ball-to-powder
 15 weight ratio, rotational speed, ball-milling time and HEAs' volume fraction in the

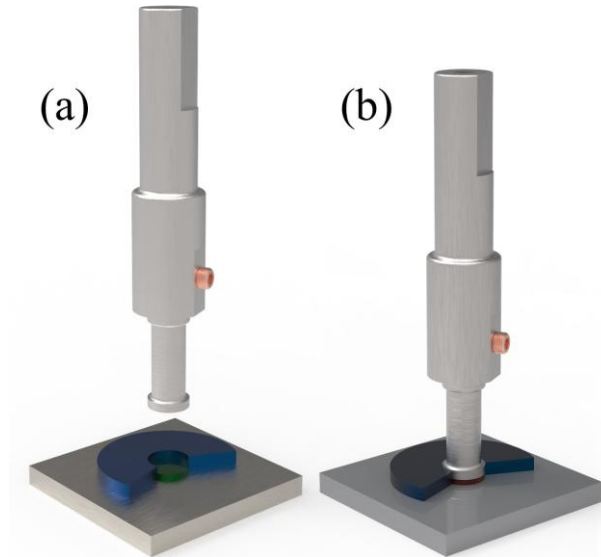
1 mixed powders were set as 60:1, 300 rpm, 5~30 hrs and 10 vol.%, respectively. Mixed
2 Al-HEAs powders after HEBM of 15 hrs, 20 hrs and 25 hrs were furtherly mixed with
3 pure Al powders to dilute the HEAs' volume fraction to 3 vol.%. Then the mixed
4 powders were ball-milled under the ball-to-powder weight ratio, rotational speed, ball-
5 milling time of 20:1, 200 rpm and 5 hrs, respectively.

6 **2.3 Equipment, procedure and process parameters of DDM**

7 Fig. 2 illustrates the schematics of DDM procedure, which are composed of the
8 pin-less cylindrical tool (made of tungsten carbide) and the concave die (made of pure
9 Al). The external diameter of the cylindrical tool and the internal diameter of the
10 concave die are both 16 mm. The cylindrical tool and the concave die were fixed on the
11 spindle of the gantry-moving type numerical control friction-stir-welding machine and
12 the workbench, respectively. The mixed Al-HEAs powders after HEBM were deposited
13 into the concave die (as shown in Fig. 2a), and the cylindrical tool started rotating,
14 plunging and holding for several seconds (as shown in Fig. 2b). During DDM process,
15 the deposited powders were rapidly solidified and continuously plasticized under the
16 effect of severe friction heat and plastic deformation heat, which leading to the
17 formation of AMCs with a certain thickness and diameter. Then the cylindrical tool
18 rotates and lifts up at the same time, and the DDM process is finished.

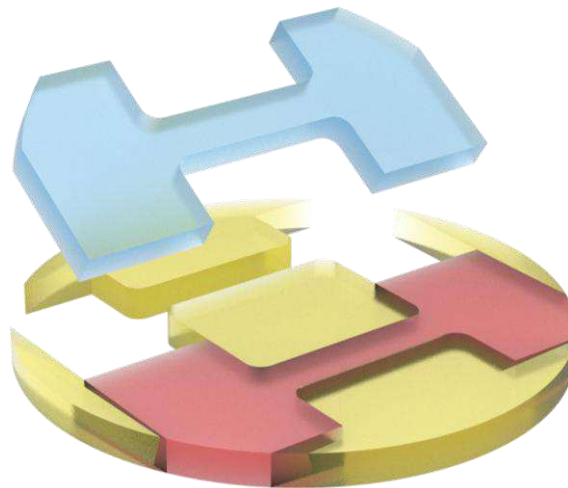
19 The rotational speed, plunging speed, plunging depth, holding time and the tilting
20 angle with respect to Z-axis of the cylindrical tool during DDM process are 400 rpm,
21 4.0 mm/min, 0.1 mm, 10 s and 0°, respectively. The thickness of the fabricated bulks is
22 set as 1.0 mm. More details about the DDM process can be referred to our published

1 researches [1]. The pure Al blocks were fabricated under the same process, including
2 ball milling and the subsequent DDM process.



3
4 **Fig. 2.** Illustrations for fabricating AMC by DDM process: (a) deposits powders into concave die
5 and (b) the tool rotates, plunges and the powders solidify into bulks.

6 **2.4 Characterization of microstructures and mechanical properties**



7
8 **Fig. 3.** Shape and the relative position of the tensile specimen respect to the DDMed blocks.

9 All the samples for the analysis of microstructures and mechanical properties were
10 perpendicular to the tool plunging direction. Scanning electron microscopy (SEM)
11 equipped with electron backscattered diffraction (EBSD) and energy dispersive

1 spectrometer (EDS) was conducted for microstructural characterization and
2 compositional analysis for powders and the fabricated bulks. Dog-bone shaped samples
3 with the gauge length of 5.0 mm, width of 1.4 mm and thickness of 1.0 mm were tested
4 in ambient tensile test. Fig. 3 illustrates the shape and the relative position of the tensile
5 specimen respect to the DDMed blocks. The tensile tests were conducted with the
6 constant speed of 0.5 mm/min.

7 **3. Results and discussion**

8 **3.1 Evolution of the mixed powders during HEBM process**

9 HEBM is considered as a kind of pre-treatment process to effectively refine the
10 particle size, of which the powder precursors are deformed, broken and refined under
11 the effect of stirring, grinding and impacting during ball milling process. The gas-
12 atomized HEAs powders possess relatively better plastic deformation capacity, thus the
13 refining effect of HEAs powders under HEBM process is limited. For the HEBM
14 process of the mixed powders that containing pure Al and HEAs, the HEAs particles
15 are supposed to be refined and uniformly-distributed in Al matrix via the cold-welding
16 effect of powders. The process parameters such as holding time during HEBM process
17 influence the evolution of the milled powders, which determine the microstructures and
18 mechanical properties of the fabricated bulks finally. Here in this paper, the pure Al
19 powders and the mixed powders of Al-10vol.%HEAs were HEBMed with the holding
20 time of 5 hours, 10 hours, 15 hours, 20 hours, 25 hours and 30 hours, respectively.

21 Fig. 4 shows the morphology of the pure Al powders under different holding times
22 during HEBM. At the initial stage of HEBM, as shown in Fig. 4a and b, the pure Al

powders were flattened into irregular flaky via the striking effect of the milling balls. Then the flaky powders were piled up into irregular polygonal powders under the effect of cold welding. With prolonging the holding time of HEBM process, as shown in Fig. 4 c and d, the piled powders were broken under the effect of strain hardening and turned from irregular polygonal particles into equiaxed particles. With further prolonging the holding time of HEBM process, as shown in Fig. 4e to h, the milled powders attained the final equilibrium state between cold welding and crushing. Thus, the morphology as well as the size of the milled powders did not change anymore.

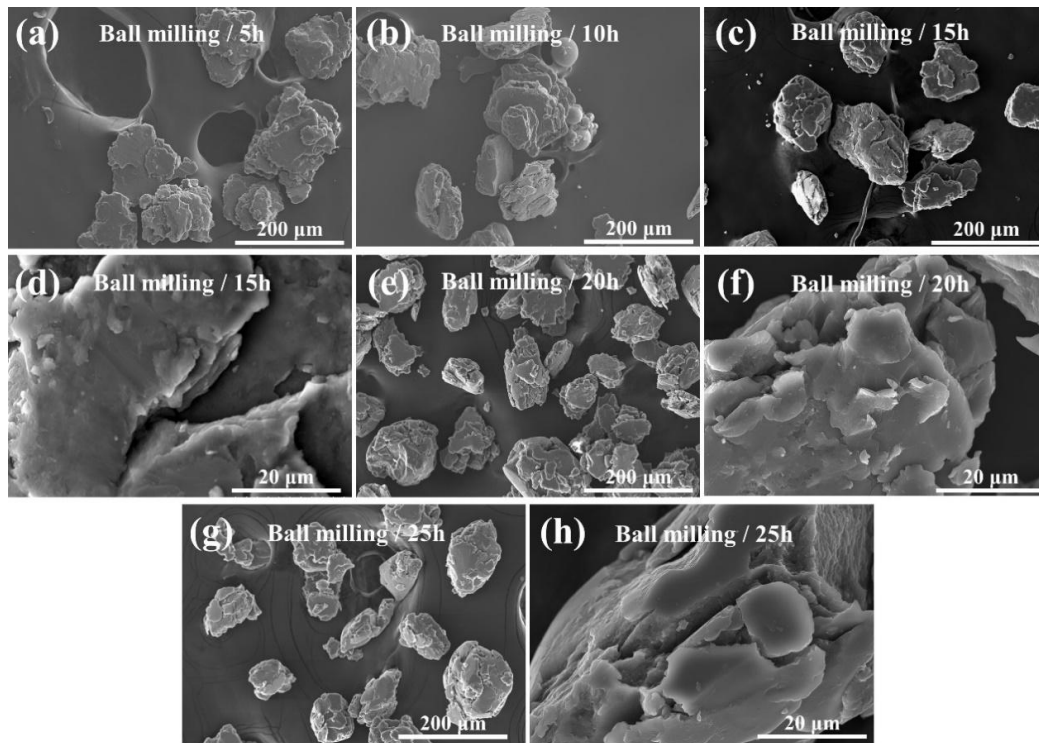
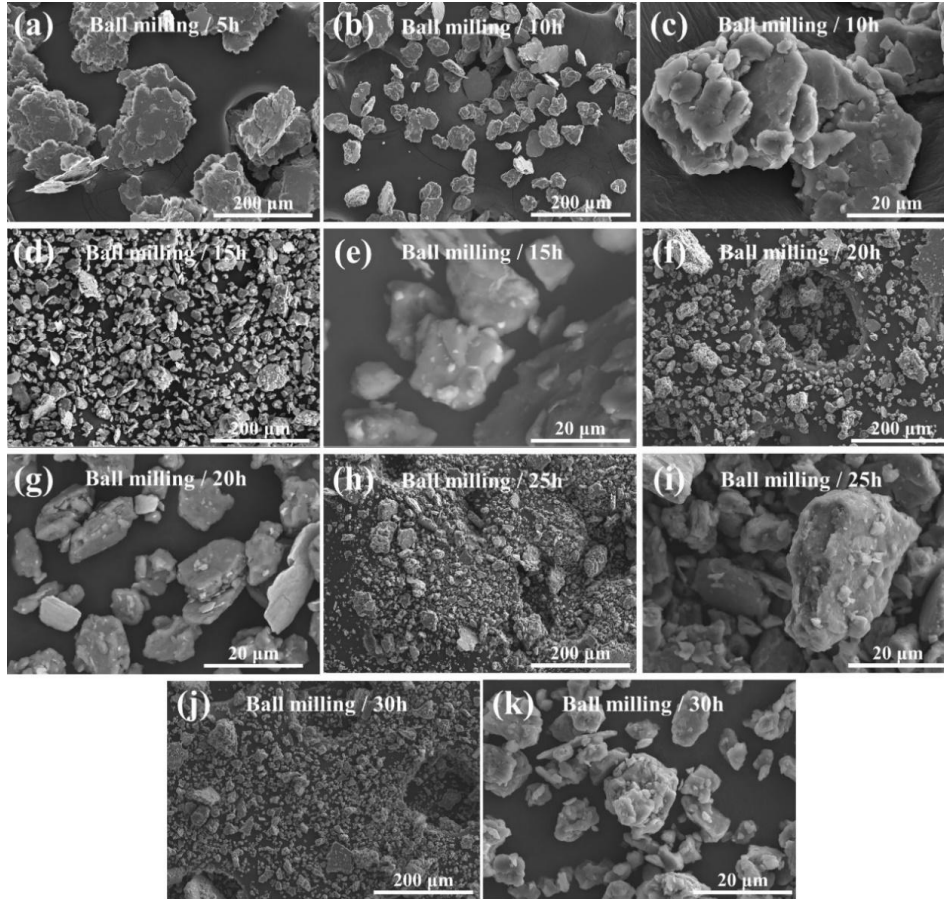


Fig. 4. Morphology of the pure Al powders under different ball milling times:
(a) 5 hrs, (b) 10 hrs, (c and d) 15 hrs, (e and f) 20 hrs and (g and h) 25 hrs.

As shown in Fig. 5, the evolution for the mixed HEAs/Al powders during HEBM process was similar with pure Al. While the incorporated HEAs powders obviously suppressed the effect of cold welding and promoted the effect of crushing, which made the final equilibrium state attained in advance. The capacity of working hardening of

1 the mixed powders was higher than that of the piled pure Al powders. The mixed Al-
2 HEAs powders were easier to be work-hardened with the decrease of toughness, as
3 shown in Fig. 5b and c, of which the effect of crushing enhanced and the effect of cold
4 welding weakened. The milled powders further crushed into fine particles with
5 prolonging the holding time of HEBM, as shown in Fig. 5d and e. As shown in Fig. 5h
6 to k, the milled powders attained the final equilibrium state between cold welding and
7 crushing under the holding time of 20 hours to 30 hours, thus the morphology as well
8 as the size of the milled powders did not change anymore.



9

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12

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Fig. 5. Morphology of the Al-HEA powders under different ball milling times:

(a) 5 hrs, (b and c) 10 hrs, (d and e) 15 hrs, (f and g) 20 hrs, (h and i) 25 hrs and (j and k) 30 hrs.

The Image-Pro Plus 6.0 software was utilized to measure and calculate the size distribution of the milled pure Al powders and the Al-HEA mixed powders under

different holding times during HEBM, as illustrated in Fig. 6. The variation for the average size of the milled pure Al powders was not obvious, of which the particle size ranged from 70~160 μm at the beginning and then decreased and stabilized at 50~130 μm , as shown in Fig. 6a. Obviously, the incorporated HEAs powders accelerated the process of HEBM via raising the effect of crushing and weakening the effect of cold welding, thus the holding time that attained the final equilibrium state between cold welding and crushing was reduced. As illustrated in Fig. 6b, the powders' size decreased quickly after the holding time of 10 hours and the average size of the milled powders maintained in the range between 5 μm and 15 μm .

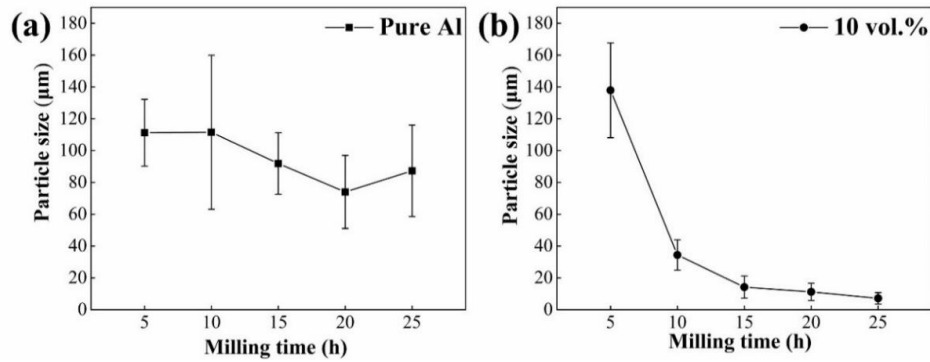


Fig. 6. Calculated particle size for pure Al and Al-HEA powders under different ball-milling time: (a) pure Al powders and (b) Al-10%HEA powders.

3.2 Evolution of the HEAs particles during HEBM

For the mixed Al-HEAs powders, the HEAs powders were involved into the pure Al powders via the formation of laminar structures during the HEBM process. The repeated cold welding and crushing of these formed laminar structures induced the crushing, refining and uniform distribution of the incorporated HEAs particles in Al powders. Fig. 7 shows the morphology of the milled powders along the cross-sectional direction under different holding times of HEBM process. At the initial stage of HEBM,

as shown in Fig. 7 a and b, the mixed pure Al powders and HEAs powders were flattened into irregular, laminar flaky and most of the incorporated HEAs particles distributed along the outside surface of the pure Al powders. With prolonging the holding time of HEBM, as shown in Fig. 7c~f, the mixed HEAs particles were severely crushed and refined. At the holding time of 25 hours during HEBM, the crushed HEAs particles distributed uniformly inside the pure Al powders with almost the similar size.

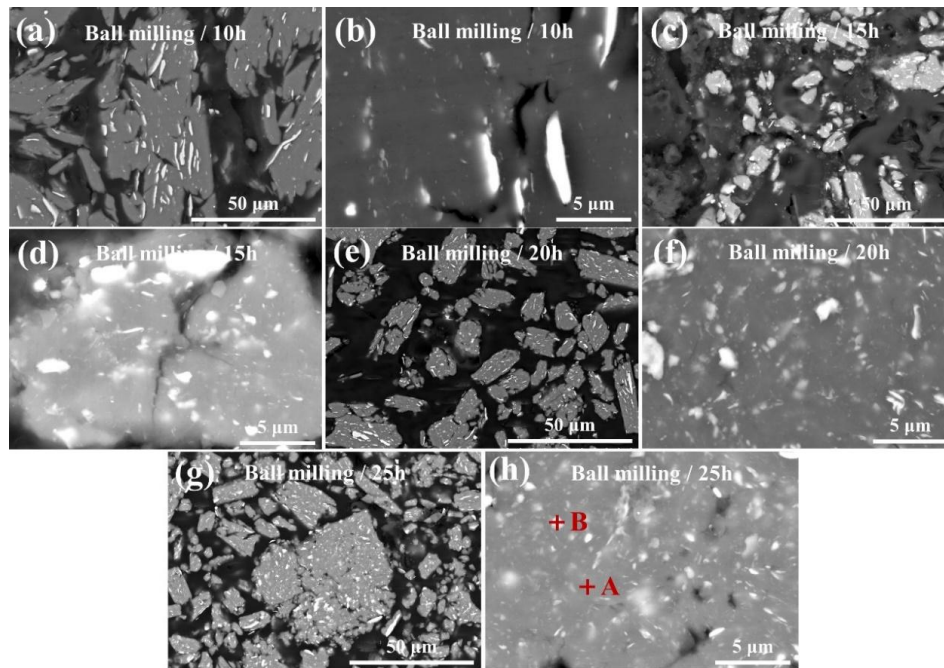
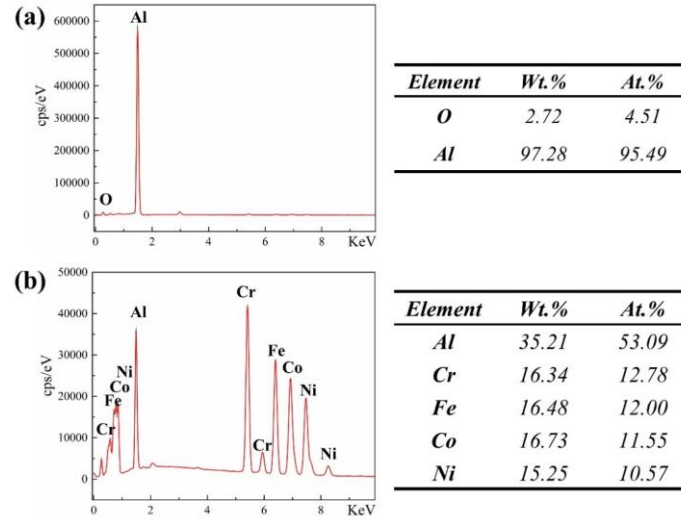


Fig. 7. Cross-sectional morphology of Al-10%HEA powders under different milling times: (a and b) 10 hrs, (c and d) 15 hrs, (e and f) 20 hrs and (g and h) 20 hrs.

Fig. 8 illustrates the elements compositions of point A and point B in Fig. 7h for the milled powders. The grey background for the point A in Fig. 7h represented the matrix for pure Al powders, and the element O in Fig. 8a may derive from oxidation film of the as-received powders, the added process control agent or the contamination during the HEBM process. The white particles for the point B in Fig. 7h represented the crushed HEAs reinforcements, of which the same content for Cr, Fe, Co and Ni indicated that the incorporated HEAs reinforcements still maintained the composition

1 stability after the holding time of HEBM for 25 hours.



2
3 **Fig. 8.** Element composition of Al-10%HEA powders in Fig. 7h: (a) point A and (b) point B.

4 Fig. 9 shows the morphology and histogram of size distribution for the HEAs
5 particles in the mixed powders after HEBM for 25 hours. As shown in Fig. 9a, a large
6 quantity of crushed and refined HEAs particles distributed uniformly in the matrix of
7 pure Al powders. Fig. 9b illustrates the histogram of size distribution of the HEAs
8 particles in the mixed powders after HEBM for 25 hours, of which the as-received
9 HEAs powders with the average size 6.5 μm turned into the equiaxed particles with the
10 average size of 0.44 μm . Besides, the size distribution of the milled powders agreed
11 with the Gaussian distribution and the ratio of the particles smaller than 0.7 μm
12 exceeded 93%, which indicated that the pre-treatment of HEBM is instrumental in the
13 refining and uniform-distribution of the incorporated HEAs reinforcements.

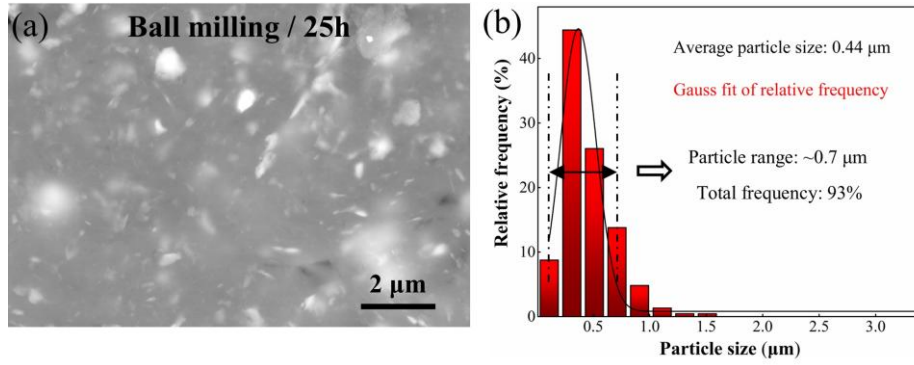


Fig. 9. (a) Morphology and (b) size distribution of HEA particles after ball milling of 25 hrs.

3.3 Morphology and distribution of the incorporated HEAs reinforcements after DDM process

The macrostructures of the fabricated composites under different holding times of HEBM were illustrated in Fig. 10, including 15 hours, 20 hours and 25 hours. It is obvious that the accumulated strain of the mixed powders during HEBM accelerated the crushing, refinement and uniform distribution of the incorporated HEAs reinforcements. When the holding time of HEBM increased from 15 hours to 25 hours, the incorporated HEAs reinforcements in Al matrix evolved from irregular polygonal particles into fine, uniformly-distributed particles, and the interfaces between the Al matrix and the incorporated HEAs reinforcements became more compact.

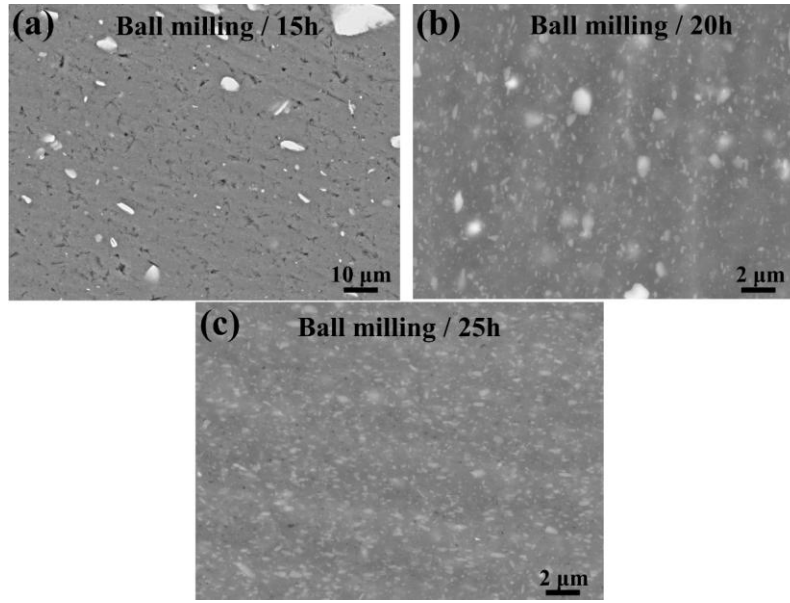


Fig. 10. Macrostructures of the fabricated composites in cross-section under different holding times during HEBM: (a) 15 hrs, (b) 20 hrs and (c) 25 hrs.

The distribution histogram of the particle size in the fabricated composites under different holding times of HEBM was shown in Fig. 11. The particle size of the incorporated HEAs reinforcements decreased significantly with prolonging the holding time of HEBM, which decreased from $1.63 \pm 1.02 \mu\text{m}$ under 15 hours to $0.11 \pm 0.07 \mu\text{m}$ under 25 hours. With prolonging the holding time of HEBM from 20 hours to 25 hours, the milled powders reached the equilibrium state between cold welding and crushing, thus there was little differences on the average particle size. Furthermore, severe circumferential-shear-deformation occurred interior the composites during the DDM process, the average particle size of the incorporated HEAs further decreased compared with the powder precursors after HEBM.

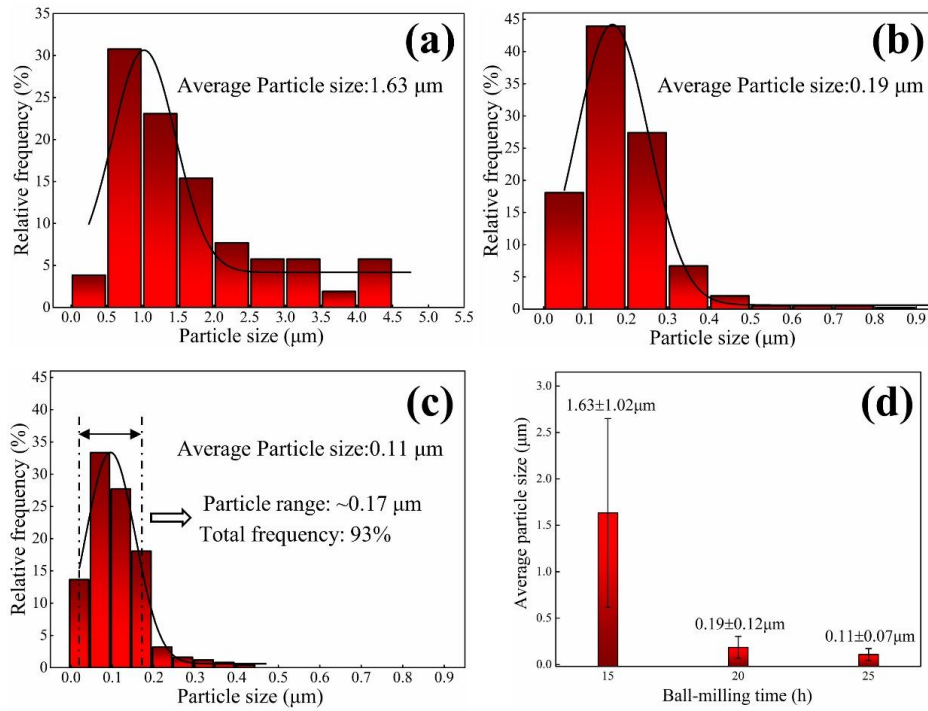


Fig. 11. Particle size distributions in composites under different HEBM times: (a) 15 hrs, (b) 20 hrs, (c) 25 hrs and (d) variation of average particle size.

3.4 Microstructures of the fabricated composites under different holding times during HEBM

Fig. 12 illustrates the inverse pole figure (IPF) images and grain size histograms of the fabricated composites under different holding times during HEBM. The average grain size of the fabricated composites first decreased and then increased, of which the average grain size for the fabricated composites under the holding time of 20 hours during HEBM was 1.16 μm . For the mixed powders that pre-treated by HEBM, the microstructural evolution of the fabricated composites was determined by both the Al matrix powders and the incorporated HEAs reinforcements. The pre-treatment of HEBM promoted the crushing, refining and uniform distribution of the mixed HEAs reinforcements, which promoted the nucleation of dynamic recrystallization and retarded the grain growth for Al matrix grains, thus leading to the refinement of the

1 recrystallized grains. Besides that, the HEBM pre-treatment increased the level of
 2 lattice-defect energy for the mixed Al powders, which was stored in the form of severe
 3 lattice distortion and high-energy dislocations. This kind of lattice-defect energy
 4 promoted the nucleation of dynamic recrystallization effectively. When the holding
 5 time of HEBM pre-treatment reached 20 hours, the crushing of the HEAs
 6 reinforcements and the accumulation of lattice-defect energy for Al powders were both
 7 instrumental in promoting the refinement of these recrystallized grains. With further
 8 increasing the holding time of HEBM, the further accumulation of the lattice-defect
 9 energy enhanced the driving force of recrystallization, which leading to the grain
 10 growth via absorbing grain boundaries and finally the increase of average grain size.

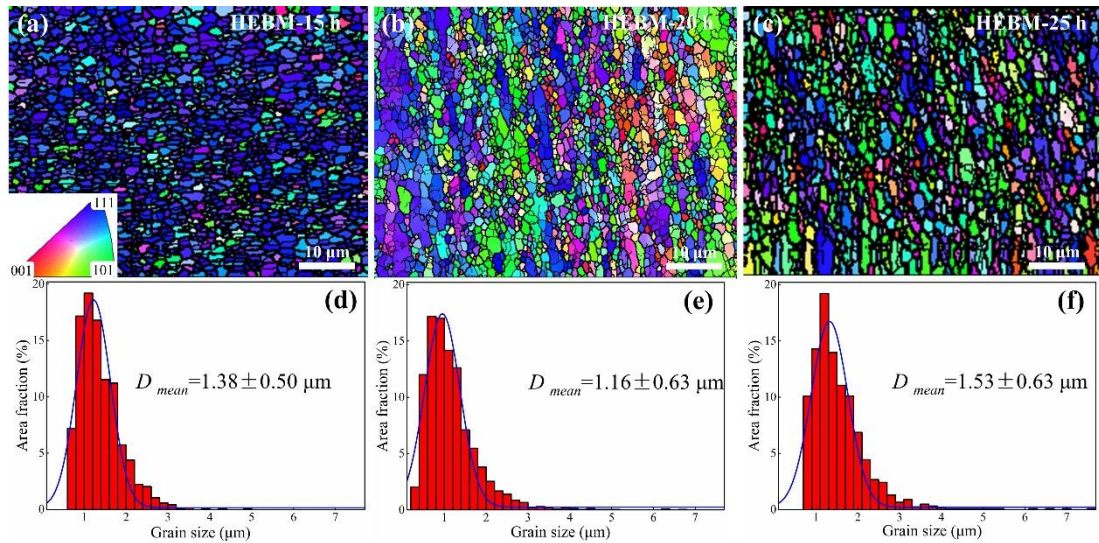
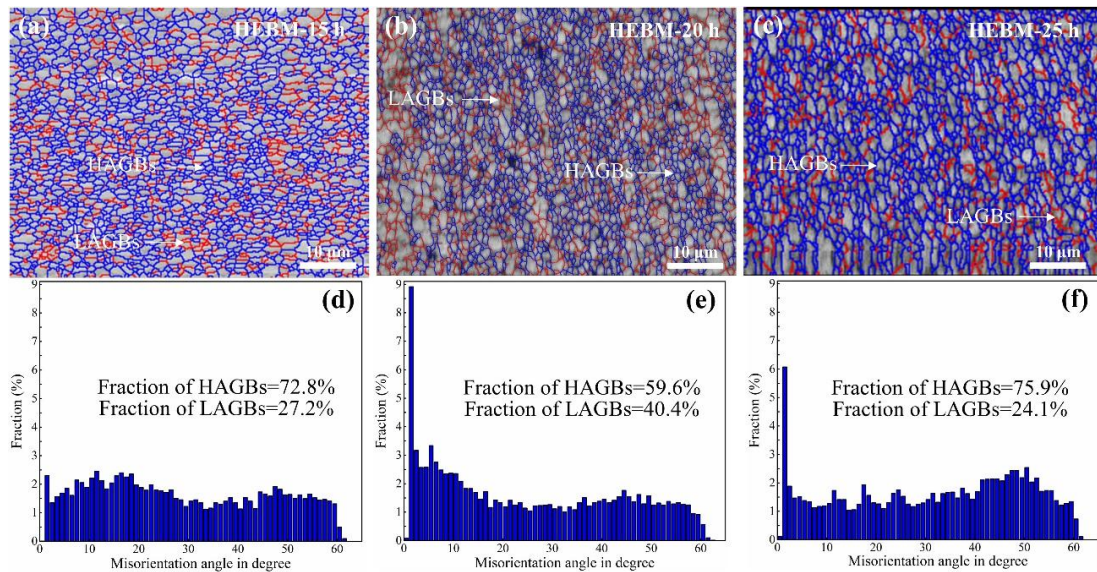


Fig. 12. IPF images and grain size histograms of the fabricated composites under different ball milling times: (a and d) 15 hrs, (b and e) 20 hrs and (c and f) 25 hrs.

Fig. 13 shows the image quality maps of the grain boundary and the corresponding misorientation angle distribution for the fabricated HEA/Al composites under different ball milling times. The blue lines are defined as the high angle grain boundaries (HAGBs, larger than 10°) and the red lines are defined as low angle grain boundaries

1 (LAGBs, ranging from 2° to 10°). The proportion of HAGBs for the fabricated
 2 composites decreased first and then increased with prolonging the holding times of
 3 HEBM pretreatment, that is, the proportion of LAGBs for the fabricated composites
 4 milled under 20 hrs was highest of all. As mentioned above, HEBM of 20 hrs
 5 accelerated the refinement and uniform distribution of HEAs reinforcements, which
 6 pinned and hindered the immigration of the LAGBs. Thus, the proportion of LAGBs
 7 for the fabricated composites milled under 20 hrs increased, as compared with the
 8 fabricated composites milled under 15 hrs. Though there were little differences on the
 9 average particle size of HEAs reinforcements, more dislocations were introduced into
 10 the Al grains with prolonging the holding time of HEBM from 20 hours to 25 hours.
 11 The density of movable dislocations interior the sub-structured grains as well as their
 12 interactions with LAGBs increased, which leading to the further increase of
 13 misorientation angle between LAGBs, thus more LAGBs turned into HAGBs.
 14 Accordingly, the proportion of LAGBs decreased and the proportion of HAGBs
 15 increased for the fabricated composites milled under 25 hrs.



16 **Fig. 13.** Image quality maps of the grain boundary and the corresponding misorientation angle
 17

distribution of the fabricated composites under different ball milling times:

(a and d) 15 hrs, (b and e) 20 hrs and (c and f) 25 hrs.

Fig. 14 shows the ratio of recrystallized grains, sub-structured grains and the deformed grains of the fabricated composites pre-treated under different holding times during HEBM. The ratio of the recrystallized grains increased and the ratio of the sub-structured grains decreased in the fabricated composites with prolonging the holding times of HEBM. The effect of plastic-deformation accumulation promoted the refinement and uniform distribution of the incorporated HEAs reinforcements with prolonging the holding times of HEBM, meanwhile the density of the restored dislocations and the content of the lattice distortion in the fabricated composites increased, which both instrumental to enhance the driving force of recrystallization and promote the recrystallization process of Al matrix. Therefore, the ratio of the recrystallized grains increased and the ratio of the sub-structured grains decreased in the fabricated composites with prolonging the holding times of HEBM.

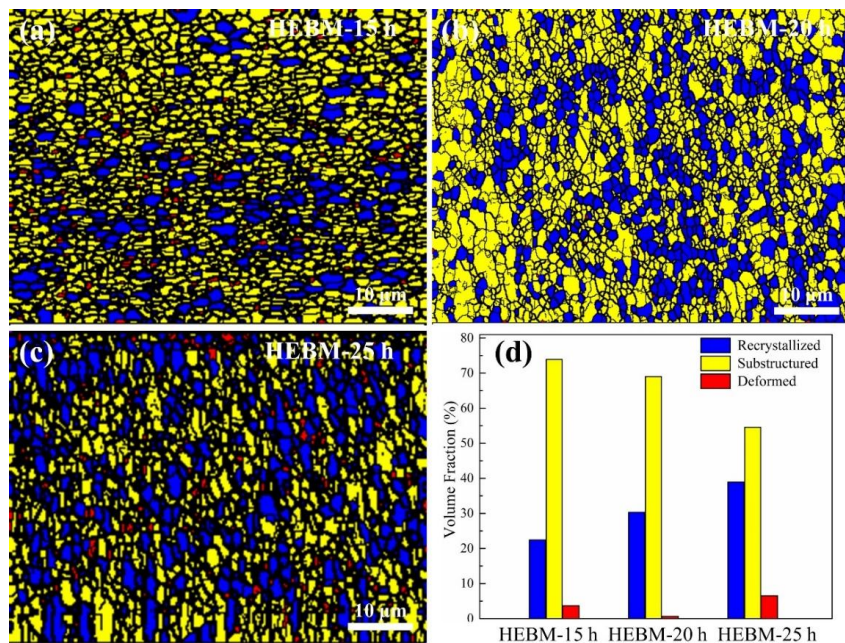


Fig. 14. EBSD maps of the recrystallized, substructured and deformed regions in the fabricated composites under different ball milling times: (a) 15 hrs, (b) 20 hrs, (c) 25 hrs and (d) statistics for

comparison.

Fig. 15 illustrates the kernel average mis-orientation maps and the angle distribution of the fabricated composites under different holding times of HEBM. It is obvious that the regions of stress concentration in the fabricated composites distributed uniformly in Al matrix. The mixed HEAs powders were broken and refined and the pure Al powders continuously accumulated strain energy with prolonging the holding times of HEBM. The uniformly-distributed HEAs reinforcements introduced massive dislocations during the DDM process, thus the values of kernel average mis-orientation for the fabricated composites continued to increase with prolonging the holding times of HEBM.

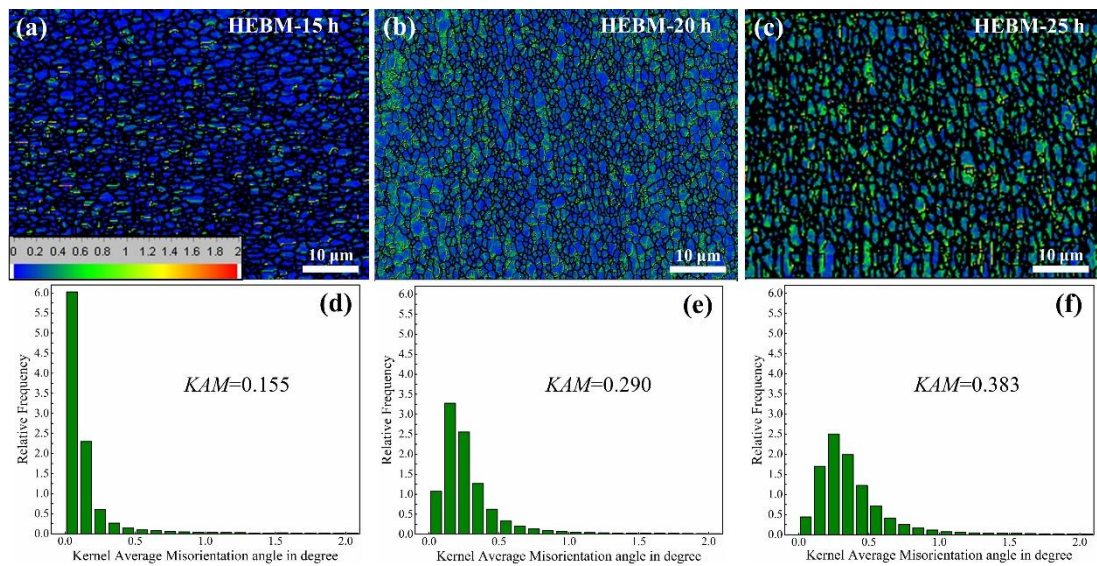


Fig. 15. Kernel average misorientation maps and the angle distribution of composites under different ball milling times: (a and d) 15 hrs, (b and e) 20 hrs and (c and f) 25 hrs.

3.5 Mechanical properties and fractography of the fabricated composites

Fig. 16 shows the mechanical properties of the fabricated Al-3vol.%HEAs composites under different holding times of HEBM pre-treatment. The yield strength and ultimate tensile strength of the fabricated composites increased while the elongation

1 decreased with increasing the holding time of HEBM process. As discussed above, the
2 HEBMed powders reached the equilibrium state between cold welding and crushing at
3 the holding time of 20 hours, of which the size of the milled powders did not change
4 anymore. Thus, the mechanical properties of the fabricated composites were similar
5 under the holding times of 20 hours and 25 hours during HEBM, of which the yield
6 strength increased from 256 MPa to 282 MPa and the ultimate tensile strength as well
7 as the elongation were both 341 MPa and 12.4%, respectively.

8 As shown in Fig. 16b, the strain hardening rates of the fabricated composites at
9 the initial stage of tensile tests increased with increasing the holding time during HEBM.
10 While the strain hardening rates of the fabricated composites under the holding time of
11 20 hours during HEBM were comparatively larger with the proceeding of tensile tests.
12 The values of strain hardening rates for composites represents the capacity of uniform
13 plastic deformation. The fabricated composites under the holding time of 20 hours
14 during HEBM possessed relatively homogenous microstructures and stress states, thus
15 the composites possessed comparatively higher ultimate tensile strength and strain
16 hardening capacity.

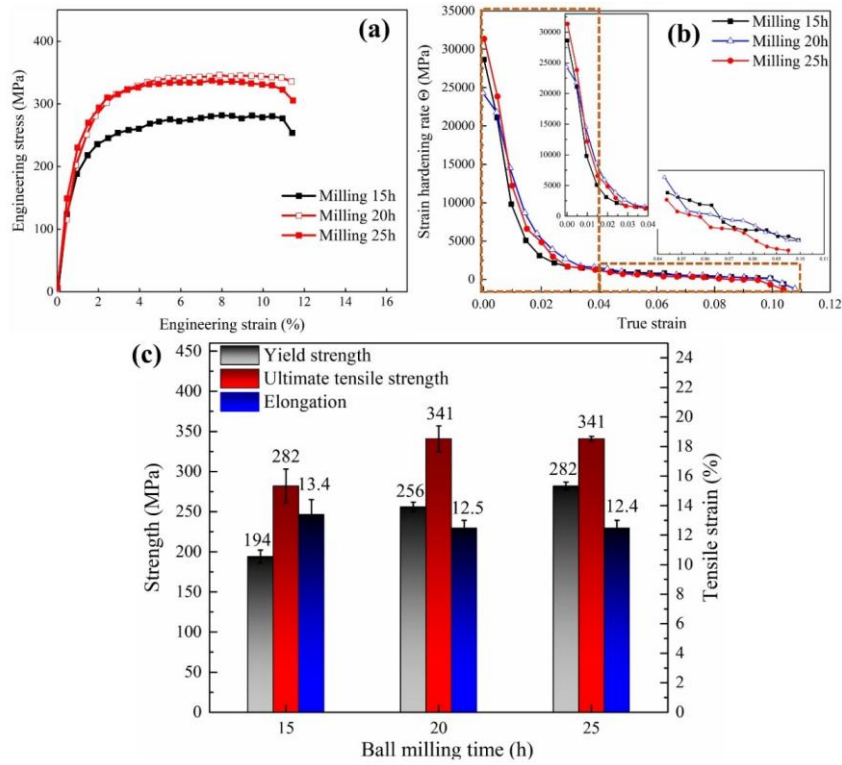
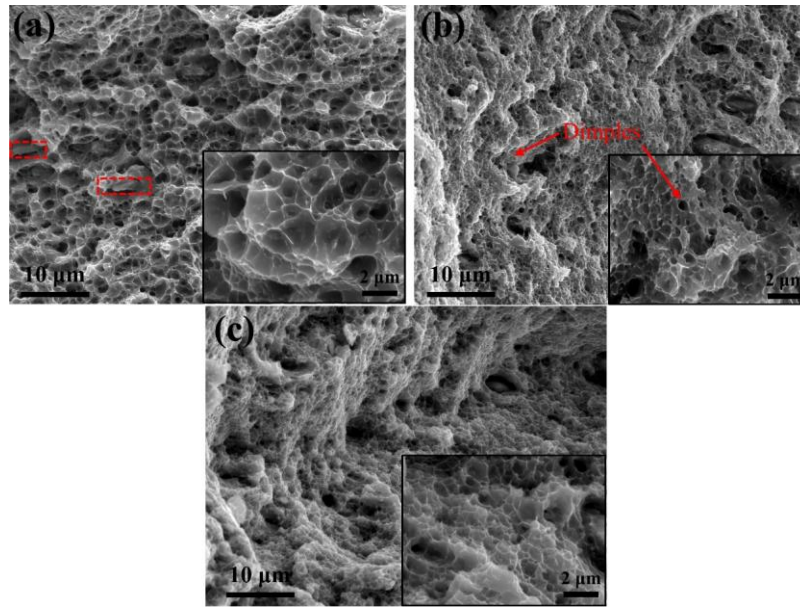


Fig. 16. Mechanical properties of the fabricated composites under different ball milling times: (a) stress-strain curves, (b) variations of strain hardening rates and (c) statistics for comparison.

Fig. 17 illustrates the fractography of the fabricated composites under different holding times of HEBM after tensile tests. The fabricated composites showed tough fracture, for which the fractography was composed of uniformly-distributed dimples. The incorporated HEAs particles in the mixed powders were refined and uniformly distributed with prolonging the holding times of HEBM, thus the interactions between the incorporated HEAs reinforcements and the internal dislocations were enhanced for the fabricated composites during tensile tests, which decreased the capacity of plastic deformation as well as the dimples' size and depth for the fabricated composites. No broken reinforcements were observed on the fracture surface for the fabricated composites under the holding time of 20 hours and 25 hours during HEBM, which indicated that the interfacial bonding between Al matrix and the incorporated HEAs reinforcements were relatively compact. When the holding time during HEBM reached

1 15 hours, as shown in Fig. 17a (as framed by red color), the HEAs reinforcements with
 2 irregular shapes emerged on the bottom of the dimples. These reinforcements with
 3 relatively larger particle size acted as the crack source to promote crack nucleation and
 4 propagation during tensile tests, thus the comprehensive mechanical properties of the
 5 fabricated composites with the holding time of 15 hours during HEBM were relatively
 6 weak, which was consistent with the stress-strain curves in Fig. 16.



7
 8 **Fig. 17.** Fractographs after tensile tests of the fabricated composites under different ball milling
 9 times: (a) 15 hrs, (b) 20 hrs and (c) 25 hrs.

10 **4. Conclusions**

11 Here in this paper, the mixed powder precursors of Al-HEAs were pre-treated by
 12 HEBM process and then fabricated into composites bulks via the subsequent DDM
 13 process. The effect of holding time during HEBM on the morphology of the mixed
 14 powders as well as the microstructures and the mechanical properties of the fabricated
 15 composites was investigated. Some conclusions can be drawn as follows:

- 16 (1) The mixed powder precursors of Al-HEAs were flattened, cold-welded, piled-up,
 17 broken and refined until reaching the final equilibrium state between cold welding
 18 and crushing during the HEBM process, and the incorporated HEAs particles

1 accelerated the effect of crushing and refinement of the mixed powders.

2 (2) With increasing the holding time of HEBM, the average particle size of HEAs
3 reinforcements in the fabricated composites decreased from 1.63 μm to 0.11 μm ,
4 and the interfacial bonding between Al matrix and the distributed reinforcements
5 improved as well as the compactness of the fabricated composites.

6 (3) The ratio of recrystallized grains increased for the fabricated composites with
7 increasing the holding times of HEBM. Besides, the yield strength as well as the
8 ultimate tensile strength of the fabricated composites increased while the
9 elongation decreased. When the holding time of HEBM reached 25 hours, the yield
10 strength, ultimate tensile strength and elongation of the fabricated composites
11 reached 282 MPa, 341 MPa and 12.4%, which proved that the pre-treatment of
12 HEBM effectively improved the strengthening effect of HEAs reinforcements.

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16 **Disclosure statement**

17 No potential conflict of interest was reported by the authors.

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