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A novel high-entropy alloy with a dendrite-composite microstructure and remarkable compression performance

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ABSTRACT

In the present study, a novel dendrite-composite high-entropy alloy consisting of nano-structured interdendritic regions and coarse dendrites was successfully designed and prepared by arc-melting. Interestingly, the as-cast Al₁₈Fe_{33.3}Co₂₃Ni₂₃C_{2.7} (at. %) alloy exhibited a great combination of high compressive strength and strain. More importantly, the new material exhibited pronounced three-stage work-hardening behavior. It was found that deformation twinning in the interdendritic region is the dominant mechanism responsible for the multi-stage work-hardening, thereby leading to good compression behavior of the new material. The synergistic effects between the dendrite and interdendritic regions are also effective for improving the mechanical property of the alloy.

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High-entropy alloys (HEAs) firstly proposed by Yeh et al. have been reported as new promising candidate materials in recent years in view of their striking performance [1–4]. At the outset, considerable research has been performed on studying single-phase HEAs. It is demonstrated that the single-phase face-centered-cubic (FCC) structured HEAs failed by the lack of the strength and body-centered-cubic (BCC) ones by the low ductility [5,6]. Recently, substantial efforts have been turned to the dual-phase or multi-phase HEAs with unique microstructures for overcoming the strength-ductility trade-off dilemma. For instance, the eutectic HEAs with a laminate structure consist of soft and hard phases [3,7,8]. The dual-phase Ni₃₅Al_{21.67}Cr_{21.67}Fe_{21.67} HEA shows a sunflower-The hierarchical nanostructured microstructure [9]. like Fe34Cr34Ni14Al14Co4 HEA is composed of BCC and B2 phases [10], and the multi-modal HEAs are prepared via mechanical alloying, followed by spark plasma sintering [11,12]. The substantial increase in strength, together with good ductility, has been observed in the above HEAs, and these alloys have gained significant attention.

Creating composite-structured materials is an important and efficient strategy to develop advanced materials with potential applications in practical engineering. In 2003, Ti-based alloys with the proper chemical composition forming the nanostructure-dendrite composite structure via controlling the casting conditions were firstly reported [13].

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Moreover, Yeh et al. [1] suggested that there exists a strong tendency to yield nanosized phases in HEAs, depending on the compositions and processing conditions. It has been reported that the spinodal decomposition as well as ordering can be easily present in HEAs, resulting in the formation of nano-scale or ultra-fine second-phase precipitates [14,15]. Up to now, nanostructures have been found in many HEAs [10,16–18], as well as in our previous work [19]. Here, a question arises on whether it is possible to design and achieve composite-structured HEAs with excellent mechanical properties by arc-melting. Based on optimizing the chemical composition, here, for the first time, we designed and reported a new multi-phase Al₁₈Fe_{33.3}Co₂₃Ni₂₃C_{2.7} HEA and achieved the nanostructure-dendrite composite microstructure by arcmelting. The detailed characterization and investigation proved that this novel alloy displayed preferable mechanical properties.

It is worth noting that the formation of the multi-phase structure is fundamental to successfully fabricate the in-situ composite microstructure. In the current work, the carbon element was selected as the trace-alloying element to increase the atomic-size difference, δ ,

($\delta = \sqrt{\sum_{i=1}^{n} c_i (1 - r / \sum_{i=1}^{n} c_i r_i)^2} \times 100$), where the c_i and r_i are the concentration and atomic radius of the ith elements, and n is the number of components, respectively. The calculated δ value of the Al₁₈Fe_{33.3}Co₂₃Ni₂₃C_{2.7} (C-doped) alloy is 8.6% within the multi-phase formation scope (δ >6.6%), whereas for the Al_{18.6}Fe_{34.2}Co_{23.6}Ni_{23.6} (Cfree) alloy, δ is 5.7%, falling into the single-phase range ($\delta \le 6.6\%$) [5,6]. Generally, the large δ will effectively facilitate the occurrence of the spinodal decomposition, and then assure the presence of the



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nanostructure in the alloy [20]. For comparison, in the present work, the microstructure of the C-free alloy was also studied.

The ingots (~30 g) of the $(AI_{18.6}Fe_{34.2}Co_{23.6}Ni_{23.6})_{100-x}C_x$ (atomic percent, at. %) alloys, where *x* is equal to 0 and 2.7, were prepared by mixing the constituent elements in a vacuum arc-melting furnace with a water-cooled Cu crucible under an argon atmosphere. Each ingot was re-melted at least six times to improve the homogeneity. Hereafter the alloys are denoted as C0 (C-free) and C2.7 (C-doped) alloys, respectively. The crystal structures and microstructures of alloys were studied, using the X-ray diffraction (XRD) with the Cu-K α radiation, field-emission scanning electron microscope (SEM, Hitachi S-4800) with backscatter electron (BSE), and transmission electron microscopy (TEM, JEM-2010) coupled with the selected area diffraction pattern (SADP). Chemical analysis was performed, employing the energy-dispersive X-ray spectrum (EDS) equipped in SEM.

For compression tests, cylinder samples with a dimension of $\Phi 3$ mm × 6 mm were prepared, using the wire-electrical-discharge machining. Then both ends of samples were polished for compression experiments. An Instron 3382 electronic-testing machine was employed for experiments at room temperature with a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. The specimens were lubricated, using graphite sheets and high-pressure grease to minimize the friction effects of both ends. The deformation strain during the compression test was measured, using an extensometer. Moreover, the machine stiffness was taken into account to determine the compression strain of samples. The detailed information to determine the deformation strain can be found in Ref. [21]. Three compression specimens (denoted as Samples 1, 2, and 3) were measured to confirm the reproducibility

of the results. In addition, post-mortem TEM characterizations were carried out to understand the deformation mechanisms of the alloy.

The XRD patterns of the as-cast CO and C2.7 alloys are displayed in Fig. 1(a) and (b), respectively. Expectedly, the baseline alloy, CO, only consists of a single BCC phase structure, while two sets of diffraction peaks corresponding to ordered-BCC (B2) and ordered-FCC (L1₂) phases are observed in the C2.7 alloy. The lattice parameters of the BCC, B2, and L1₂ phases are calculated to be 0.2874 nm, 0.2887 nm, and 0.3707 nm, respectively. Fig. 1(c) and (d) show the SEM-BSE images of the as-cast alloy. It suggests that the CO alloy [Fig. 1(c)] exhibits a single-phase feature, which is in good agreement with the XRD result [Fig. 1(a)]. By contrast, the C2.7 alloy [Fig. 1(d)] presents the typical dendrite microstructure displaying alternate bright [interdendritic (ID)] and dark [dendrite (DR)] regions. Interestingly, a close view of the local microstructure in the ID region reveals that substantial nanosized cuboidal particles are uniformly distributed in this region, as shown in the inset of Fig. 1(d). The volume fractions of the DR and ID regions in the C2.7 alloy measured from >10 SEM-BSE images are verified to be approximately 48% and 52%, respectively. The macroscopic SEM-EDS point analysis results of the DR and ID regions in the current C2.7 alloy given in Table 1 show that the Al and Ni elements in the dendrites are higher than those in the ID region, while those of Fe, Co, and C are enriched in the ID region. The SEM-EDS mapping analysis was also performed to map the elemental distributions in the DR and ID regions, and the results are given in the Fig. S1 (see the Supplementary Material). Similarly, it shows that Al and Ni are enriched in the DR region, while Fe, Co, and C are preferentially segregated in the ID region. This trend is due to the fact that the pairs of Fe-C and Co-C have the higher negative



Fig. 1. XRD patterns and SEM-BSE images of the as-cast Al₁₈₆Fe₃₄₂Co_{23.6}Ni_{23.6} alloy (a) and (c), Al₁₈Fe_{33.3}Co₂₃Ni₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂₃Co₂₃Ni₂Co₂₃Ni₂₃Co₂₃Ni₂₃Co

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Table 1

Chemical compositions (at. %) of the DR and ID regions in the as-cast $Al_{18}Fe_{33.3}Co_{23}Ni_{23}C_{2.7}$ alloy. Note that there exists a large error about the result of the carbon content due to the micro-content of carbon and the limitation of the current system to detect light elements.

Region	Al	Fe	Со	Ni	С
Norminal DR region ID region	$\begin{array}{c} 18 \\ 26.8 \pm 0.57 \\ 14.1 \pm 1.25 \end{array}$	$\begin{array}{c} 33.3 \\ 23.3 \pm 0.26 \\ 38.1 \pm 0.24 \end{array}$	$\begin{array}{c} 23 \\ 20.5 \pm 0.29 \\ 26.6 \pm 0.23 \end{array}$	$\begin{array}{c} 23 \\ 27.5 \pm 0.22 \\ 16.5 \pm 0.62 \end{array}$	$\begin{array}{c} 2.7 \\ 1.9 \pm 3.68 \\ 4.7 \pm 2.85 \end{array}$

enthalpy of mixing (-50 kJ/mol and -42 kJ/mol, respectively), compared to other pairs [22]. It can be concluded that there exists elemental segregation during solidification, which is beneficial to the formation of multimodal microstructures [23].

In order to further identify the microstructure of the C2.7 alloy, TEM and SADP were conducted. Fig. 2(a) shows the hybrid microstructure consisting of DR and ID regions of the alloy and the corresponding SADP result [the inset of Fig. 2(a)] of the dendrites. Fortunately, the dendrites present the B2 single-phase coarse structure, while nano particles distribute regularly in the ID region. The detailed nature of the ID region was presented in Fig. 2(b-d). It is obvious that nano-sized cuboidal particles with sizes of 40–100 nm are uniformly embedded in the matrix. This trend is in good agreement with the SEM analysis, as shown in the inset of Fig. 1(d). In line with the SADPs obtained from the particles [Fig. 2(c)] and matrix [Fig. 2(d)], it indicates that the matrix is an FCC structure, while the particles are the superlattice L1₂ structure. This fascinating morphology is notably similar to that reported in Ni-based superalloys, i.e., the coherent ordered L1₂ particles were uniformly dispersed in the FCC matrix [24]. Hence, the as-cast alloy actually consists of three phases, i.e., B2, FCC, and L1₂ structures. The lattice parameters of these phases are calculated from SADPs, being, respectively, 0.2875 nm, 0.3792 nm, and 0.3779 nm. Note that it is difficult to distinguish the FCC phase from the $L1_2$ phase in the XRD pattern [Fig. 1(b)], since it has a similar lattice parameter with the L1₂ phase. The lattice misfit, ε , between the FCC and L1₂ phases can be calculated, using the lattice-mismatch formula, i.e., $\varepsilon = 2 \times (a_{L1_2} - a_{FCC})/(a_{L1_2} - a_{FCC}) = -0.34\%$ within the range of -0.2% to -0.5% of the Nibased alloy [24]. It can be confirmed that in the ID region, the L1₂ nano particles have the fully-interfacial coherence with the FCC matrix, which contributes favorably to its high strength and good ductility. Therefore, the present alloy can be characterized by a novel composite microstructure consisting of the nano-scale ID region and micro-scale dendrites. Fig. 2(e), which was drawn by the image software Adobe Photoshop CS6 and based on the SEM image of the alloy, shows the schematic of this novel dendritecomposite structure clearly. Even though the B2/L1₂ nano particles have been often found in previous reported HEAS [10,18,19], it is believed that this is the first time to report the in-situ dendritecomposite microstructure in HEAs.

Representative compression engineering and true stress-strain curves with the strain rate $(5 \times 10^{-4} \text{ s}^{-1})$ of three tested samples of the as-cast C2.7 alloy are presented in Fig. S2(a) (see the Supplementary Material). We select the Sample 2 as an example, here, to discuss the compression behavior of the alloy. As shown in Fig. 3(a), obvious yielding and work-hardening are evident, and the alloy exhibits a high yield strength (~1223 MPa) and reasonable fracture strain (~27.8%) together with the significant compressive strength (~3396 MPa) for the engineering stress-strain curve. While the yield strength, fracture strength, and strain are 1202 MPa, 2448 MPa, and 32.6% respectively, for the true strain-stress curve (black line). In Fig. 3(b), under the true strain-stress condition, a comparison of the compressive strength versus strain-to-failure of the Ti-based dendrite-composite alloys [23,25] and early reported HEAs [5,26–28] as well as some HEAs with unique microstructures [8,9] is presented. It is evident that the as-cast Al₁₈Fe_{33.3}Co₂₃Ni₂₃C_{2.7} alloy exhibits a better combination of the compressive strength and plasticity than any other alloys. This remarkable mechanical performance is usually attributed to the unique dendrite-composite microstructure, i.e., the composite effect of the DR and ID regions [25]. Expectedly, the fracture morphologies (Fig. S3 in



Fig. 2. TEM image of the as-cast Al₁₈Fe_{33.3}Co₂₃Ni₂₃Co_{2.7} alloy (a), and the magnified image of the ID region (b), and the schematic of the nanostructure-dendrite composite structure (e). Note that this schematic was drawn by the image software Adobe Photoshop CS6, based on the SEM image of the alloy. The corresponding SADPs of the DR [the inset of (a)], and ID region (c) and (d). Superlattice spots are indicated by green circles.



Fig. 3. (a) Room-temperature compressive curves of the as-cast $Al_{18}Fe_{33.3}Co_{23}Ni_{23}C_{2.7}$ alloy tested at a strain rate of 5×10^{-4} s⁻¹, (b) maximum strength versus strain for the dendritecomposite high-entropy alloy, compared to some other Ti-based composite alloys [23,25], and early reported high-entropy alloys [5,8,9,26–28] in the true strain-stress condition, and (c) the variation of the strain-hardening rate (red solid curve) and true stress (black dash line) versus true strain of the alloy.

the Supplementary Material) of the dendrite-composite material demonstrate the typical fractography of the dendrite-composite materials. A detailed description of the fractography is in the Supplementary Material.

In order to further understand the mechanical behavior of the alloy, we have carefully studied the work-hardening rate (Θ) , which is commonly used to characterize the plastic-deformation behavior of alloys. The work-hardening rate was investigated by differentiating the true stress over strain [29], $\Theta = d\sigma_T/d\varepsilon_T$, where σ_T and ε_T are the true stress and strain, respectively. Fig. 3(c) presents the work-hardening curve of the Sample 2, where we just take into account the post-yielding behavior of the alloy. Evidently and interestingly, the strain-hardening curve of the alloy shows notable three different regimes during the compression test at the constant strain rate of 5×10^{-4} s⁻¹. Fig. S2 (b) (see the Supplementary Material) depicts the strain-hardening curves of the tested three samples. It's clear that all of the three samples present the remarkable three-stage work-hardening behavior. The Stage 1 can be characterized by the decrease of the strain-hardening rate. After Stage 1, the strain-hardening rate increased with increasing the strain (Stage 2), followed by a progressive decrease of the strainhardening rate with a further increase in the strain (Stage 3). It is generally believed that this distinguishable three-stage work-hardening behavior can be interpreted in light of the combined change in the deformation mechanisms [30,31]. In the following section, TEM characterizations are carried out to specifically investigate the deformation mechanisms of the dendrite-composite HEA, which can help explain its exceptional mechanical behaviors.

The mechanical behavior is closely associated with the deformation mechanism. Thereby, the evolution of the deformation structure was investigated at several deformation stages [true strains of 5.6% and 12.8%, corresponding to the Stages 1 and 2 in Fig. 3(c), respectively] by TEM for further verifying the underlying deformation mechanisms of the alloy. Fig. 4(a) and (b) depict the microstructure of the alloy after deformation at a true strain of 5.6%. It is clear that substantial dislocations occur in the DR region and aggregated at the DR side near the DR/ID boundary. Moreover, one can clearly see dislocation accumulations in the FCC matrix of the ID regions, as depicted by the green arrows in Fig. 4(b). It is reasonable that the dislocation-based plasticity mechanisms for the coarse dendrites and the FCC matrix of the nano-structured ID region are responsible for the initial work-hardening behavior (Stage 1) of the alloy. As described in Fig. 4(c) and (d), interestingly, extensive deformation twins (as a deformed substructure) occurred in the ID region of the sample, which was subjected to further deformation at a true strain of 12.8% (Stage 2 in Fig. 3c). Meanwhile, it can be seen clearly that the deformation twins cutting through the nano-cuboidal L1₂ particles, similar to the published study that the mechanical twinning shearing the nano-scale γ' -particles during the deformation process of Ni-based alloys at 725 °C [32]. Such mechanical twinning associated with an increase of the work-hardening rate during deformation of metals, which has been suggested by previous literatures [33,34] as



Fig. 4. Representative TEM images of samples subjected to compression true strains of 5.6% (a-b) and 12.8% (c-d): (a) dislocations within the dendritic and interdendritic regions as well as aggregated at the DR/ID boundary, (b) dislocation accumulation (green arrows) in the FCC matrix of the ID region, (c) the bright-field image of the ID region, and (d) an enlarged dark-field image in (c) showing that high-density deformation twins occurred in the ID region.

well as been supported by the $Al_{0.1}$ CoCrFeNi and $Al_{0.3}$ CoCrFeNi HEAs under compression tests [31,35]. After Stage 2, in the ID region, deformation-twinning evolution gradually decreases with a further increase in the strain, and dislocation-slip becomes the dominant deformation mechanism, resulting in a decrease of the work-hardening rate in Stage 3 [31,36]. This change of the dominant deformation mechanism, from the dislocation-movement in the dendrites at Stage 1 to the deformation-twinning in the nano-structured ID region at Stage 2, could be used to further reasonably explain the multi-stage work-hardening behavior [in Fig. 3(c)] of the alloy under compression at room temperature. As the result of the strong hardening ability, the compressive strength and plasticity revealed a good combination in our novel dendrite-composite HEA, as found in Fig. 3(a).

Finally, the remarkable strength-ductility combination under compression of the dendrite-composite HEA is related to the unique multistage work-hardening behavior caused by the multiple mechanisms operated under deformation. Including the dislocation, deformation twinning, etc., in particular, with the complex composition (Al, Fe, Co, Ni, and C), multi phases (FCC, L1₂, and B2 structures) form to provide the micro-structural heterogeneity. In conventional polycrystalline materials [37], the work-hardening rate under compression either shows a monotonous decrease with increasing the strain or is constant in Stage 2, whereas the rate increases unusually with increasing the strain in Stage 2 in our novel dendrite-composite high-entropy alloy. It's worth mentioning that the mechanical behaviors of the novel HEA are determined by the volume fractions and intrinsic properties of the DR and ID regions. As referred by previous studies on the multiphase Al_{0.7}CoCrFeNi HEA [38,39]. The intrinsic length scale of the BCC region (comprising ordered B2 and disordered BCC phases) [38] as well as the FCC-BCC interface [39] also has significant effects on the mechanical properties of the alloy. In the present case, the overall deformation behavior of the new material results from the interaction of the DR and ID regions. The ongoing work is performed to investigate the detailed influences of the intrinsic length scale of the ID region and the interface between the DR and ID regions on the nanostructure-dendrite composite HEA by means of the nanoindentation tests, which is out of the scope of the present work.

In summary, a novel dendrite-composite HEA, Al₁₈Fe_{33.3}Co₂₃Ni₂₃C_{2.7}, was designed and prepared by arc-melting. The as-cast alloy exhibits a bimodal microstructure comprising of the coarse DR (B2) and nanostructured ID regions (L1₂ nano-scale particles coherently embedded in the FCC matrix). It was found that in the true strain-stress condition, the as-cast alloy presents the ultra-high fracture strength (2448 MPa) along with a reasonable strain (32.6%) as well as high compressive yield strength (1201 MPa). The remarkable compression behavior of the alloy is clearly mediated by the multi-stage work-hardening behavior due to dislocation movements in the DR region and the FCC matrix of the ID region, and deformation twinning in the ID region. The interaction between the dendrite and interdendritic regions has beneficial effects on the mechanical behavior of the alloy.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi. org/10.1016/i.scriptamat.2018.09.008.

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