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Paper Title:A new lightweight Al2.7TiVCrCu high entropy alloy with excellent strength and toughness after homogenization treatment

Content :

Results

The design value of HEA content and the actual value measured by ICPAES are shown in Table S1 (Appendix A). The total content of HEA changes slightly. The design value is $Al_{40}Ti_{15}V_{15}Cr_{15}Cu_{15}$, and the actual value is $Al_{41}Ti_{16}V_{15}Cr_{13}Cu_{15}$. For convenience of explanation, it is still recorded as $Al_{2.7}$TiVCrCu-HEA. Fig. 1 (a) is the schematic diagram of multi-stage melting of $Al_{2.7}$TiVCrCuHEA, and Fig. 1 (b) and (c) show the backscattered electron image measured by EPMA. The specific content of each phase is shown in Table 1. The XRD diagram of cast and annealed $Al_{2.7}$ TiVCrCu HEA is shown in Fig. 1 (d). Fig. 1 (e) is a selected area electron diffraction (SAED) pattern containing all four phases calibrated by TEM.

Discussion

From the composition diagram Fig. 1, it is clear that the $Al_{2.7}$TiVCrCu HEA mainly consists of four phases before and after annealing, namely α, β, γ and θ. The tissue morphology in the as-cast state displays a dendritic shape, and after annealing the dendritic crystals disappear significantly and the tissue morphology displays an island shape. The specific content of each phase is shown in Table 1. The content of the constituent elements of α, θ and γ phases remains basically unchanged after annealing. While in the β phase, the contents of Cr and V increased significantly, and Al, Ti and Cu all decreased to different degrees [28].

As seen in Fig. 1(d), the positions of the XRD peaks remained unchanged after annealing, while the full width at half maximum of the peaks became much narrower compared to the as-cast condition, indicating that the annealing treatment caused growth of the grains. Analysis using Jade software showed that HEA has XRD peaks with BCC, FCC and HCP structures. T To further determine the phase structure, a region containing four phases on the annealed sample was cut for TEM observation using the FIB technique and selected area electron diffraction (SAED) was performed. The results in Fig. 1(e) demonstrate that the α, β, γ, and θ phases are FCC, BCC, FCC, and HCP structures, respectively, which is also in general agreement with the XRD results.

After homogenization annealing, the average grain size increased from 26 μm to 84 μm and the morphology changed from dendritic to island-like, as shown in Fig. 2. The volume fraction of BCC phase decreases from 51% to 45%, the volume percentage of FCC phase increases from 43% to 50%, and the HCP phase remains basically around 5–6%. The mapping results show that all phase elements of the same phases are uniformly distributed without significant separation, which is consistent with the results in Table 1. Each phase is present in the form of a randomly oriented distribution.

The hardness and Young's modulus were estimated using Olivier's method combined with the load-displacement curves. In this experiment, the holding time was set to 5 s. The γ-phase was not tested because its area was too small and beyond the accuracy of the nanoindentation test. The nano-hardness of each phase was tested four times and the average value was taken. The hardness of all three phases decreased after annealing. The modulus of α and θ phases decreased slightly, while the modulus of β phase increased slightly, which may be due to the variation of elemental content in β phase. The highest hardness and modulus were found for the θ phase of the HCP structure with 9.93 GPa and 199.99 GPa, respectively, and 7.88 and 197.79 GPa after annealing. The lowest was found for the α phase of the FCC structure, but it still reached 6.48 and 171.91 GPa. The local elastic recovery of the indentation was positively correlated with the ratio of hardness to elastic modulus [29].

When the ratio is low, the indentation after unloading will be deeper, and the local energy dissipation will be higher, which indicates the characteristics of low brittleness. The table in Fig. 4 (b) shows that the H/E values of all phases decrease after annealing, which further proves that the toughness of the material increases after annealing. Among them, the H/E values of the θ phase are much smaller than those of the other phases. Due to discontinuous yielding in plastic deformation, many serrations appear on the displacement unloading curve in Fig. 3 (a), which is caused by the interaction between moving dislocations and diffusing solute atoms, resulting in transient negative strain rate sensitivity with local changes variations [30,31].

After homogenization annealing, the compressive strength increases from 1330 MPa to 1805 MPa, and the fracture strain increases from 18% to 32%, while the yield strength basically remains unchanged as shown in Fig. 3 (c). Generally, the increase of grain size will lead to the reduction of grain boundaries and the reduction of dislocation barriers, which will lead to the increase of plasticity and the decrease of strength of the material [32].

However, the experimental results of this work show that not only the toughness has been improved, but also the compressive strength has been significantly improved, which can be attributed to the coordinated deformation behavior between the soft phase (FCC) and the hard phase (BCC and HCP). The FCC phase exhibits extraordinary work-hardening ability through planar dislocation slip and laminar dislocation deformation. After yielding, the strain-hardening indices calculated by curve fitting in the strain-strengthening phase (elliptical region in Fig. 3(d)) are 0.52 and 0.96, respectively. The strain hardening exponent of the $Al_{2.7}TiVCrCu$ HEA increased after annealing, indicating the increase of its resistance to uniform plastic deformation.

There are many cracks within the θ (HCP) phase in Fig. 4(a), but it is difficult for the cracks to extend into the adjacent soft phase because the soft phase coordinates the deformation of the surrounding grains and inhibits crack extension during the deformation process. The HCP phase has high hardness and low H/E value, and as mentioned above, the local energy dissipation in this phase is high and the cracks do not extend outward, which ensures the overall stability and strength. When the stress is high enough, the crack crosses the HCP phase boundary and gradually expands and fractures.

Fig. 4(c) shows that the average grain size becomes smaller after deformation, which is about 53 μm. The grains undergo lattice rotation after compression and change from large-angle grains to small-angle grains, with an average grain orientation difference of 17°. Small-angle grain boundaries account for a certain proportion, especially the frequency of grain boundaries in the 5° range is high, which indicates the presence of many deformed grains inside the material, while 5–15° sub grain boundaries also account for a certain proportion. Although not dominant, large-angle grain boundaries also exist to varying degrees. Both sub grain boundaries and large-angle grain boundaries absorb dislocations and reduce dislocation density.

Fig. 4 (d) shows that the plastic deformation of FCC structural phase is the largest, followed by BCC, and the deformation of HCP structural phase is small. The average dislocation density after deformation is significantly higher than that before deformation. Therefore, the change of microstructure after annealing causes the hard phase to be surrounded by the soft phase, and the soft phase and hard phase deform in a coordinated manner, which improves the strength and toughness of the high entropy alloy. Fig. 4 (e) shows that most HEAs have high strength but poor fracture strain, or high fracture strain with low strength [[33], [34], [35], [36], [37]]. Surprisingly, $Al_{2.7}TiVCrCu$ HEA with multiphase structure has excellent strength and toughness combinations, as well as low density, which is promising for applications.

5. Conclusions

In summary, a lightweight $Al_{2.7}TiVCrCu$ HEA was prepared by using the multi-stage melting method without changing the elemental components and ratios. After annealing at 1273 K for 24 h, the HEA was endowed with excellent strain-hardening ability, and both strength and toughness were effectively improved, with compressive strength reaching 1805 MPa and fracture strain reaching 32%. The results indicate that high-temperature annealing leads to a change in the elemental content of the BCC phase and a decrease in the volume fraction. The FCC phase and the HCP phase both remain unchanged in composition and increase in volume fraction to 51% and 6%, respectively. The FCC phase coordinates the deformation of surrounding grains and inhibits crack extension, and this coordinated deformation behavior between soft and hard phases ensures the overall strength and toughness. These results may provide a valuable reference in overcoming the strength-ductility mismatch in high-entropy alloys.