Paragraph Ran in the Queries

Paper Title: Enhancing fatigue resistance of high-entropy alloy by designing a hierarchically heterogeneous microstructure

Content :

Tensile behaviors

Fig. 3(a) shows engineering and true stress-strain curves of present HEA. This HEA demonstrates good combination of yield strength (~870 MPa), ultimate tensile strength (~1060 MPa) and ductility (~26 %). The strength-ductility synergy of present HEA is superior to that of most reported $Al_{0.3}CoCrFeNi$ HEAs in previous studies. Complex microstructure leads to joint effect of multiple strengthening mechanisms, which is responsible for high yield strength [24]. Namely, quasi-ultrafine-grained boundaries, TBs, B2 precipitates and residual dislocations serve as barriers to prevent dislocation motion, producing refinement grain, precipitation and dislocation strengthening effects. In addition, back-stress strengthening induced by heterogeneous microstructure is another important strengthening mechanism, which has been frequently reported in previous studies [25].

On the other hand, the reason for good combination of ultimate tensile strength and ductility was clarified from following aspects. First, a high density of dislocations were found within such fine grains after tensile fracture (Fig. 3(b)), indicating good dislocation storage ability associated with low SFE. It is known that high dislocation storage enhances work hardening, which is beneficial for strength and ductility [26]. Second, it is seen that dislocations closely interact with pre-existing deformation twins in PRG region, as shown in Fig. 3(c). Deformation twins can hinder dislocation movement and simultaneously accommodate more dislocations in twinning planes. The role of deformation twins in improving work-hardening ability has been frequently reported in HEAs with low SFE [27]. Last but not least, the strain incompatibility between hard and soft domains during deformation induces the pile-up of geometrically necessary dislocations at soft/hard domain interface in heterogeneous grain microstructure (i.e., RGs, PRGs and OGs), contributing to back-stress strengthening and hardening effects and eventually enhancing work-hardening ability. In addition, the existence of B2 precipitates with different morphologies, sizes and distributions also creates strain gradient and back stress, promoting above effects [10]. To sum up, the good work-hardening ability, which is related to complex microstructure as analyzed above, delays the occurrence of necking and fracture, leading to a good combination of ultimate tensile strength and ductility.

Fatigue deformation and crack initiation

In the following, we attempted to clarify fatigue crack initiation mechanisms, mainly based on deformation and damage morphologies, in order to provide an explanation for excellent fatigue property of present HEA. Fig. 5 reveal fatigue cracking features under different cyclic stress levels. Several parallel bands with spacing of ~2 μm emerged in non-recrystallized regions adjacent to the heterogeneous interface (see blue line), however, no deformation and damage features were detected in recrystallized regions. Obvious extrusions/intrusions are derived from these bands. Above band morphologies are similar to those during fatigue in nanotwinned strengthened 316L stainless steel consisting of nanotwinned and recrystallized grains [33], in which these bands were called persistent slip band (PSB)-like shear bands. Since shear bands are subjected to large local plastic strain, fatigue cracks are formed in these weak sites, thus indicating shear band-mediated fatigue cracking mechanism. Fatigue crack growth terminates when encountering the interface, which is attributed to the variation of strength and plasticity in both sides of interface.

In order to determine the origin of PSB-like shear bands, fatigue deformation was investigated through TEM method. It is seen that cyclic loading produces a high density of dislocations in deformation twins of PRG regions, as shown in bright-field images (Fig. 6(a) and (c)). Corresponding dark-field images (Fig. 6(b) and (d)) show that a large amount of B2 precipitates were distributed along TBs, which is consistent with above observation. Elastic deformation mismatch between B2 precipitates and matrix as well as stress concentration induced by B2 precipitates facilitates the formation and motion of dislocations. However, fine twin lamellae serve as the obstacles to dislocation motion and then induce dislocation accumulation. In this case, local plastic deformation during cyclic loading is accumulated in the form of dense dislocations against deformation twins decorated with B2 precipitates. The evolution of dense dislocations in local region leads to the formation of PSB-like shear bands and fatigue crack initiation.

It is well known that fatigue is a strain and damage localization process [34]. In metal materials, fatigue cracks usually initiate from PSBs. As analyzed above, there mainly exist RGs and PRGs in present HEA. Quasi-ultrafine RG scale and small precipitates within grains suppress the activation of slip deformation, preventing the generation of PSBs. It seems that PRGs can provide the sites for nucleating PSBs due to large grain size of tens of microns. However, deformation twins, precipitates and residual dislocations as well as back stress induced by heterogeneous microstructure can strengthen this type of grains. High strength suppresses the nucleation of PSBs and then leads to the formation of shear bands, causing shear band-mediated crack initiation.

In addition to PSBs, interfaces usually serve as the stress concentrators to cause fatigue crack initiation. Dislocation accumulation at precipitate interfaces produces stress concentration, which may lead to the development of microcracks [35]. B2 precipitates in present HEA possess a semi-coherent Kurdjumov–Sachs *(K–S)* relation with matrix material [36]. In addition, the size of B2 precipitates is far lower than micron scale. Above two characteristics will lessen dislocations pile-up at the interfaces, decreasing the possibility for interfacial cracking. On the other hand, it is reported that fatigue crack initiation occurs at heterogeneous interfaces between non-recrystallized and recrystallized regions in partially recrystallized metals after thermo-mechanical treatment [34]. This phenomenon was not found in present HEA after observing surface damage morphologies of most fatigue fractured specimens. The heterogeneous interfaces between PRG and RG regions exhibit an enhanced resistance to fatigue cracking, probably due to good cyclic deformation compatibility between two regions which alleviates strain localization at interfaces [33].

Microstructural instability including grain coarsening and detwinning during fatigue was reported in ultrafine-/nano-grained and nanotwinned metals [37,38]. Fatigue deformation and damage are prone to generate from these local inhomogeneities, deteriorating the fatigue property. As mentioned above, present HEA includes the microstructures of fine grains and deformation twins. However, a large amount of B2 precipitates are pinned along the grain and twin boundaries, suppressing the occurrence of grain coarsening and detwinning after fatigue to fracture. The good microstructural stability during fatigue is beneficial for the improvement of the resistance to fatigue crack initiation.

Fatigue crack propagation

Besides fatigue crack initiation mechanisms, fatigue crack propagation behaviors were explored to further understand fatigue property. Fig. 7(a) exhibits the feature of a propagating fatigue crack with the length of ~100 μm. It is seen that main fatigue crack path is torturous with several deflection points. Several segments of this crack were magnified for a detailed observation. Fig. 7(b) reveals that when main crack reaches non-recrystallized region, two inclined cracks are bifurcated from main crack tip. One crack proceeds along inclined shear band formed in non-recrystallized region; another crack extends along heterogeneous interface between recrystallized and non-recrystallized regions. Crack deflections along the interfaces between two regions were also detected in other locations of main crack, as shown in Fig. 7(c) and (d). In our previous studies, it was found that the fatigue crack deflected along inclined slip bands ahead of crack tip in homogeneous coarse-grained structure of same composition [16], which was distinctly different from crack deflection behavior in present heterogeneous structure. Fig. 7(e) and (g) reveal fatigue crack features in recrystallized regions. A large amount of precipitates are embedded into crack path. These precipitates can locally change crack growth direction and eventually produce a slightly rough crack path. In addition, a second microcrack was found along interface between recrystallized and non-recrystallized regions