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Unexpected cyclic stress-strain response of dual-phase high-entropy alloys induced by partial reversibility of deformation

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ABSTRACT

The recently developed dual-phase high-entropy alloys are characterized by pronounced strain hardening and high ductility under monotonic loading owing to the associated transformation induced plasticity effect. Fatigue properties of high-entropy alloys have not been studied in depth so far. The current study focuses on the low-cycle fatigue regime. Cyclic tests were conducted and the microstructure evolution was studied post-mortem. Despite deformation-induced martensitic transformation during cycling at given plastic strain amplitudes, intense strain hardening in the cyclic stress-strain response is not observed. This behavior is attributed to the planar nature of slip and partial reversibility of deformation.

The development of new alloys combining high strength with excellent ductility has stimulated numerous research activities in recent years, mostly focusing on steels. High-Mn steels have led to considerable advances in recent years [1–5]. Through compositional tuning, the stacking fault energy (SFE) can be tailored for two deformation effects, namely, twinning induced plasticity (TWIP) and transformation induced plasticity (TRIP). Depending on the actual value of the SFE, being affected by chemical composition and temperature, either mechanical twinning or martensitic transformation are prevalent besides planar dislocation slip [2,3,5,6–9]. Dislocation-twin and dislocation-phase boundary interactions lead to high strain hardening reserves and delayed necking under monotonic loading [1–5,10–13].

Recently, interest has increased in similar strain hardening effects observed in high-entropy alloys (HEAs). Primarily, these alloys, also referred to as multi-component alloys or compositionally complex alloys, are composed by at least five elements in equiatomic composition [14]. This concept is different from earlier alloying philosophies which typically use only one dominant base element [1–13]. Due to this principal difference in the alloy design approach also non-equiatomic alloys consisting of only four main elements are in the literature often referred to as HEA such as the alloy studied here [15].

Mainly two groups of HEAs have been characterized quite comprehensively, viz. refractory HEAs and HEAs composed by group-IV transition elements [16,17]. Within the latter group the well-known single phase Cantor-alloy, i.e. Fe-Mn-Ni-Co-Cr, has been studied in some detail, particularly focusing on the deformation behavior and fracture toughness at cryogenic temperatures [18–20]. Deformation in the Fe-Mn-Ni-Co-Cr system is characterized by dislocation slip and twinning, the latter becoming more prevalent in the cryogenic regime [18,20]. Thus, some similarities between the behavior of high-Mn TWIP steels and the Fe-Mn-Ni-Co-Cr HEA are obvious [21–23]. The material studied here is a $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ alloy, characterized by a two phase microstructure obtained after thermo-mechanical processing including quenching from final annealing temperature [15]. Upon tensile straining the metastable face-centered cubic (fcc) phase shows a martensitic transformation to the ε-hexagonal closed packed (hcp) martensite phase. Following monotonic deformation to a local engineering strain of about 65%, the fraction of martensite increases to about 85%. Concomitantly, intense strain hardening is observed. Detailed analysis of the microstructure revealed stacking faults, dislocations patterns, mechanical twins and ε-martensite [15].

These results document the rapid progress in alloy development regarding better understanding and tuning of the monotonic deformation behavior of HEAs. However, when in service, engineering alloys are usually not loaded monotonously but in a non-monotonic fashion. Yet, currently no data reporting on the cyclic behavior and concurrent microstructure evolution in HEAs in the low-cycle fatigue (LCF) regime are available. Only three studies reporting on $Al_{0.5}CoCrCuFeNi$, tested under four-point bending fatigue loading [24,25] and $Al_{0.1}CoCrFeNi$, also tested under bending loading [26], provide first insights into this important topic. All these studies focus on the fatigue strength of the alloys and, thus, on the high-cycle fatigue (HCF) regime, as is also emphasized in current reviews reporting on HEAs [17,27,28]. Generally, in LCF

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and HCF regimes different loading conditions are considered, i.e. strain and stress controlled testing, respectively, and, thus, microstructure evolution is significantly different.

From high-Mn steels it is known that microstructure evolution under either monotonic or cyclic loading can differ significantly from each other [29–32]. Some of the current authors studied microstructure evolution in various fatigue regimes ranging from LCF to fracture mechanical testing [30,31]. Interestingly, the TWIP steels probed in these studies, i.e. thermo-mechanically processed micro-alloyed Fe-Mn22-C0.6, did not reveal twinning under cyclic loading [31]. Only re-arrangement of dislocation structures was observed under cyclic loading at room temperature (RT) [31]. Thus, counter-intuitively, high accumulated strains led to cyclic softening instead of cyclic hardening as would have been expected from the monotonic deformation behavior. Only monotonic pre-deformation, prior to cyclic loading, was able to stabilize fatigue response due to increase in twin density and, thus, intensified twin-dislocation interactions [31].

The current study aims at providing first insights into the microstructure evolution of dual-phase HEAs under cyclic loading. Testing at RT in the LCF regime is accompanied by microstructure characterization employing X-ray diffraction and electron microscopy. The results reveal an unexpected microstructure upon testing: despite significant evolution of strain induced martensite, hardly any associated strain hardening is observed during cycling up to about 90,000 cycles at low strain amplitude of $\Delta\varepsilon/2 = 0.23\%$ and 10,000 cycles at high strain amplitude of $\Delta\varepsilon/2 = 0.6\%$, i.e. accumulated plastic strains of well above 1000% in all cyclic tests conducted. The ε-martensite fraction of about 95 vol% developing upon fatigue at the within this study studied highest cyclic strain amplitude (0.6%) even exceeds values found upon monotonic testing. The results are discussed based on the underlying deformation characteristics.

The initial ingot was cast in a vacuum induction furnace using pure metals and subsequently thermo-mechanically processed [15]. Two different conditions are probed here. A *fine grained* dual-phase condition has been obtained by annealing at 900 °C followed by water quenching. The second one, referred to as *coarse grained* condition has been subsequently heat treated at 850 °C for 20 min followed by furnace cooling. The coarse grained condition shows hardly any hcp phase in the initial state, i.e. well below 1 vol% according to EBSD and non-detectable in the XRD spectrum (cf. Figs. 1 and 3). Initial martensite fraction for the fine grained condition is about 3 vol% according to EBSD analysis. Samples with gauge section dimensions of 8 mm × 3 mm × 1.5 mm were cut by electro-discharge machining (EDM). In order to remove the affected surface layer all samples were subsequently mechanically ground and polished down to 5 μm grit size. Furthermore, samples were vibration polished using conventional oxide polishing suspension (OPS). Mechanical tests were conducted using a MTS load-rig in fully reversed push-pull loading in strain control. For determination of strains a miniature extensometer featuring a 3 mm gauge length was directly attached to the specimen. Nominal strain rate in all tests was $6 \times 10^{-3}\ \mathrm{s}^{-1}$. For phase analysis an X-Ray diffractometer (XRD) equipped with a Cu Kα source operated at 40 kV was used. Microstructure analysis including electron channeling contrast imaging (ECCI) was done using a high-resolution scanning electron microscope (SEM) at acceleration voltage of 30 kV. The SEM employed is equipped with an electron backscatter diffraction (EBSD) unit and a backscattered electron (BSE) detector.

Fig. 1 shows initial microstructures and the cyclic stress responses of the $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ alloy in both conditions in the LCF regime. Most pronounced evolution of microstructure is expected in the LCF regime. Thus, first fatigue tests in the dual-phase TRIP-HEA were conducted under strain controlled conditions at low to relatively high strains. Currently, no results regarding HCF performance of the TRIP-HEA are available, however, will be provided in future studies, amongst others focusing on the role of plastic strain amplitude, strain rate and testing frequency, respectively. Strain amplitudes in current work ranged from $\Delta\varepsilon/2 = 0.23\%$ to $\Delta\varepsilon/2 = 0.6\%$. In order to prevent buckling of the miniature samples the load was increased stepwise during the very first cycles and the final strain amplitude was reached after 50 to 75 cycles depending on the strain amplitude. Thus, the initial cycles are not shown in Fig. 1 for the sake of clarity. The fine grained and the coarse grained TRIP-HEA conditions are characterized by an almost stable stress plateau throughout the tests. Minor strain hardening is only observed for relatively high strain amplitudes. For the strain amplitudes of $\Delta\varepsilon/2 = 0.23\%$ and $\Delta\varepsilon/2 = 0.28\%$ even slight softening is observed in the coarse grained TRIP-HEA (Fig. 1b). As can be seen in Fig. 1a, the fine grained TRIP-HEA is characterized by stable response at the small strain amplitudes (upon initial transient behavior, which cannot be evaluated due to the initial minor loops conducted for avoiding buckling), while for $\Delta\varepsilon/2 = 0.4\%$ and $\Delta\varepsilon/2 = 0.6\%$ slight hardening sets in. Stress amplitudes at a given strain amplitude are higher for the fine grained TRIP-HEA, which is due to the smaller initial grain size. In all cyclic tests for the fine grained and coarse grained conditions the accumulated plastic strain was well above 1000%. The absence of pronounced strain hardening upon cyclic loading as compared to the case of monotonic testing up to local engineering strain of about 65%, where intense strain hardening is observed [15], is striking and will be discussed below. Most importantly, despite the pronounced martensitic transformation found, intense dislocation-phase boundary interactions seem not to be present. From numerous studies focusing on a huge variety

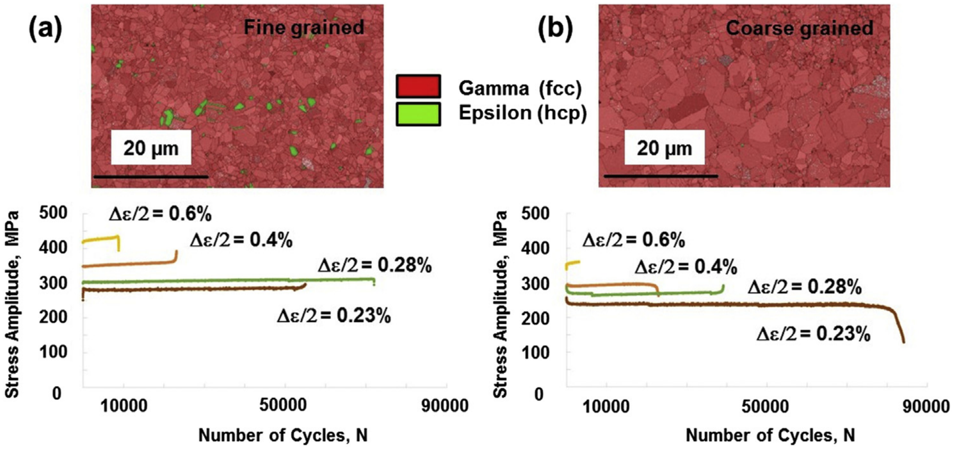

Fig. 1. Cyclic stress response of (a) fine grained (about 5 μm average grain size) and (b) coarse grained (about 10 μm average grain size) TRIP-HEAs tested under various strain amplitudes at room temperature and constant strain rate. The EBSD phase maps highlight differences in the initial microstructural conditions (red: γ-phase, green: ε-phase). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)