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In-situ SEM observation of phase transformation and twinning mechanisms in an interstitial high-entropy alloy

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ABSTRACT

The recently developed interstitial high-entropy alloys (iHEAs) exhibit an enhanced combination of strength and ductility. These properties are attributed to dislocation hardening, deformation-driven athermal phase transformation from the face-centered cubic (FCC) γ matrix into the hexagonal close-packed (HCP) ε phase, stacking fault formation, mechanical twinning and precipitation hardening. For gaining a better understanding of these mechanisms as well as their interactions direct observation of the deformation process is required. For this purpose, an iHEA with nominal composition of Fe-30Mn-10Co-10Cr-0.5C (at. %) was produced and investigated via *in-situ* and interrupted *in-situ* tensile testing in a scanning electron microscope (SEM) combining electron channeling contrast imaging (ECCI) and electron backscatter diffraction (EBSD) techniques. The results reveal that the iHEA is deformed by formation and multiplication of stacking faults along {111} microbands. Sufficient overlap of stacking faults within microbands leads to intrinsic nucleation of HCP ε phase and incoherent annealing twin boundaries act as preferential extrinsic nucleation sites for HCP ε formation. With further straining HCP ε nuclei grow into the adjacent deformed FCC γ matrix. γ regions with smaller grain size have higher mechanical stability against phase transformation. Twinning in FCC γ grains with a size of ~10 μm can be activated at room temperature at a stress below ~736 MPa. With increasing deformation, new twin lamellae continuously nucleate. The twin lamellae grow in preferred directions driven by the motion of the mobile partial dislocations. Owing to the individual grain size dependence of the activation of the dislocation-mediated plasticity, of the athermal phase transformation and of mechanical twinning at the different deformation stages, desired strain hardening profiles can be tuned and adjusted over the entire deformation regime by adequate microstructure design, providing excellent combinations of strength and ductility.

1. Introduction

High-entropy alloys (HEAs) attract high attention as they tap a sheer infinite elemental composition space, offering opportunities for discovering novel properties and their combinations [1–15]. The recently developed interstitial high-entropy alloys (iHEAs) open this pathway even further owing to the huge leverage that small elements on inter-lattice sites render on constitution and properties, as is also known from many steels. More specific, some iHEAs exhibit enhanced combinations of strength and ductility, due to activation of practically all possible strengthening effects, e.g. interstitial and substitutional solid solution strengthening, twinning-induced plasticity (TWIP), transformation-induced plasticity (TRIP), composite effects, precipitation hardening, work hardening by dislocations and stacking faults as well as grain refinements [12,16]. The mixing entropy pertaining to the Fe-30Mn-10Co-10Cr-0.5C (at. %) iHEA (studied in this work) is relatively low compared to those HEAs with more than 5 principal elements in equimolar fraction [1,4,16,17]. The authors noted that the maximized configurational entropy is not the sole factor determining phase stability of HEAs. Indeed, previous work of the current authors on the development of novel TRIP-assisted dual-phase HEAs has been motivated by the facts that first, both enthalpy and entropy together determine alloy stability and second, the absence of detrimental intermetallics is in some cases a more essential boundary condition for successful alloy design than entropy maximization alone [4,17]. Our main point is thus more placed on arriving at a stacking-fault energy dependent point of phase

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metastability for obtaining more superior mechanical properties through the associated athermal deformation driven transformation mechanisms.

Among these aforementioned deformation mechanisms, mechanical twinning is the most extensively studied effect [8–15]. The activation of mechanical twinning has been reported to effectively enhance strain hardening of HEAs at cryogenic, i.e. liquid nitrogen temperature [11,13]. The characterization techniques mainly employed so far in that context included electron backscatter diffraction (EBSD), transmission electron microscopy (TEM) and neutron-diffraction based techniques. EBSD allows mapping of the evolution of crystallographic features as a result of mechanical twinning, e.g. local and global grain orientations, kernel average misorientations and geometrically necessary dislocations (GNDs) at a wide field of view [12,13,15]. By employing TEM, the formation of stacking faults (SFs), nucleation and growth of twinning are revealed [8,10]. By employing *in-situ* neutron diffraction, the evolution of mechanical twinning was evaluated based on the evolution of the stacking fault probability [9].

In contrast to mechanical twinning, phase transformation in HEAs is less commonly studied [5–7]. Synchrotron X-ray diffraction (SXRD) and TEM have been employed to characterize phase transformation products in HEAs. While the influence of grain orientation on deformation-driven phase transformation can be studied by SXRD [5] and the transformation products can be revealed by dark-field diffraction in TEM [6,7], the transformation kinetics associated with these phenomena are still unknown. In addition, deformation-driven phase transformations are rapid and diffusionless. This means that in HEAs more detailed *in-situ* tracking of the nucleation and growth of deformation-driven phase transformation products during deformation is a pending challenge.

Yet, for designing HEAs and iHEAs with enhanced mechanical properties, detailed understanding of all active deformation mechanisms and their interaction is essential. Particularly the magnitude of the stacking fault energy (SFE), which is mainly controlled by chemical composition and temperature, is an important factor controlling activation of these multiple coexisting and competing deformation mechanisms [18–22]. For avoiding possible effects of compositional inhomogeneity on the SFE and the associated deformation mechanisms in different samples, it is therefore preferable to sequentially map microstructure evolution in the same region, i.e. via *in-situ* testing. Since microstructure evolution is then monitored in the same material portion, *in-situ* straining also allows to better reveal direct links among dislocation and partial dislocation activities, mechanical twinning and phase transformation. However, such insights, when obtained from deformation of thin foil specimens exposed to *in-situ* TEM probing, are sometimes not fully transferable to bulk materials owing to foil and surface related mechanical boundary condition effects. Also, TEM reveals microstructure features in a relatively confined region. Similar arguments hold for *in-situ* SXRD/neutron diffraction methods due to low direct spatial microstructure correlation. Some of these shortcomings can be overcome by probing microstructure, dislocations and crystallographic texture using *in-situ* high resolution scanning electron microscopy (SEM) [23–26]. By using bulk samples, *in-situ* high resolution SEM is capable of unraveling deformation mechanisms in bulk materials. Due to its large field-of-view and high spatial resolution (down to ~80 nm) [27], interrupted *in-situ/in-situ* EBSD based on high-resolution SEM has proven itself as a powerful tool to study phase transformations in many systems, e.g. multi-phase TRIP steels [25,26,28], quench and partitioning steels [29,30], and high-Mn austenitic steels [31,32]. As a SEM-based tool with high spatial resolution, e.g. ~2.9 nm using an acceleration voltage of 30 kV and a beam current of 11.6 nA, electron channeling contrast imaging (ECCI) has been successfully applied to the observation of dislocations, partial dislocations and mechanical twinning in many systems [28,33–37]. Here we therefore use the combination of *in-situ* and interrupted *in-situ* joint EBSD and ECCI probing in the SEM as a wide field of view method for directly observing activation of dislocations, stacking faults (SFs), mechanical twinning and displacive phase transformation with the aim to unravel the strain hardening mechanisms in a iHEA.

2. Experimental procedures

The nominal chemical composition of the iHEA is Fe-30Mn-10Co-10Cr-0.5C (at. %). The alloy was cast in a vacuum induction furnace, hot rolled and homogenized at 1200 °C for 2 h, followed by water quenching. The alloy was cold rolled to ~60% total thickness reduction in several rolling passes and then annealed at 900 °C for 3 min under Ar atmosphere, and eventually quenched in water.

To reveal the elemental distribution and characterize the microstructure, energy-dispersive X-ray spectroscopy (EDX), EBSD and ECCI probing in the SEM were employed. Samples were prepared by grinding, polishing in diamond suspension and final polishing in colloidal silica suspension for ~1.5 h. The last step ensures that the sample surface is deformation-free. EDX mapping was conducted in Zeiss-Merlin at 15 kV. EBSD measurements were carried out at 15 kV with a step size of 80 nm using a JEOL JSM-6500F instrument. For ECCI measurements, a Zeiss-Crossbeam XB 1540 FIB-SEM and a Zeiss-Merlin instrument were used each operated at an acceleration voltage of 30 kV.

To track the phase transformation and correlate it with the deformation substructure, interrupted *in-situ* EBSD and ECCI observations of the same sample regions were conducted, consisting in the repeated observation of the same material portions during interrupted tensile testing at different strain levels. Deformation was imposed as a uniaxial tensile load on dog-bone samples at room temperature with an initial strain rate of 10^{-3}/s using a Kammrath and Weiss tensile stage. After deforming the sample to pre-defined strain levels, it was unmounted and transferred to SEM for microstructure characterization.

To additionally track the evolution of microstructure during deformation without interrupting loading, *in-situ* tensile testing was conducted using a Zeiss-FIB SEM with a home-built tensile stage. Prior to the *in-situ* tensile test, EBSD measurements were carried out on polished sample in the undeformed state. Then ECC images of same regions were taken both in the undeformed state and after different strain levels without unloading the sample. All strain values obtained both, during the interrupted *in-situ* and the *in-situ* tests were calculated by measuring the local length changes of the investigated regions along the loading direction.

3. Results

3.1. Microstructure in the undeformed state and mechanical properties

The microstructure and elemental distribution in the undeformed state are shown in Fig. 1. SEM EDX mapping reveals homogeneous distribution of all elements at the grain scale (Fig. 1b–e). Thus, a specific influence of compositional inhomogeneity on the phase transformation and twinning kinetics can be excluded. The true stress-strain and strain hardening curves are shown in Fig. 1f. Since true strain is calculated from engineering strain following the constraint of constant volume, no true strain can be calculated anymore after necking, thus the data is presented up to the end of uniform deformation (prior to necking). The iHEA