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Influence of deformation and annealing twinning on the microstructure and texture evolution of face-centered cubic high-entropy alloys

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ABSTRACT

The influence of the physical mechanisms activated during deformation and annealing on the microstructure and texture evolution as well as on the mechanical properties in the equiatomic CoCrFeMnNi high-entropy alloy (HEA) were investigated. A combination of cold rolling and annealing was used to investigate the HEA in the deformed, recovered, partially recrystallized, and fully recrystallized states. Detailed microstructure and texture analysis was performed by electron backscatter diffraction, transmission electron microscopy, and X-ray diffraction. The mechanical properties were evaluated using uniaxial tensile testing. A specific focus of this investigation was put on studying the influence of deformation and annealing twinning on the material behavior. It was substantiated that during cold rolling deformation, twinning facilitates the transition from the Cu-type to the Brass-type texture, whereas annealing-twinning leads to a strong modification of the texture formed during recrystallization. The formation of specific twin orientations and the randomization of the recrystallization texture were proven by experiments as well as by cellular automaton simulations. During tension of the cold-rolled and annealed material high work-hardenability was observed. We attribute this behavior primarily to the dominance of planar dislocation slip and reduced tendency for dynamic recovery, since deformation twinning was observed to activate only in the non-recrystallized grains and thus, contributed minimally to the overall plasticity. The correlation between deformation/annealing twinning and the microstructure evolution, texture development, and mechanical properties was discussed in detail.

1. Introduction

High entropy alloys (HEAs) are a new family of metallic materials, which are characterized by the alloying of more than 4 elements in usually equiatomic or near-equiatomic compositions. Despite the complex chemical composition of these alloys, they crystallize in simple crystal structures and form non-ordered solid solutions. The reason for this unique behavior and the idea behind HEAs is that significantly high mixing entropies increase the stability of the solid solution against the formation of intermetallic compounds [1]. These alloys were first introduced in separate publications by Cantor et al. [2] and Yeh et al. [3] and have rapidly gained the interest of the materials science community owing among others to their exceptional mechanical properties. In fact, HEAs are characterized by unusual combinations of properties not found in conventional alloys, such as high hardness, strength, and wear resistance, exceptional fracture toughness at cryogenic temperatures, and high corrosion and oxidation resistance [4–6]. The cause of these exceptional properties stems from different effects that influence the response of the materials to external stimuli. Besides the high-entropy effect that stabilizes the solid solution, also inherently slow diffusion kinetics, a severe lattice distortion, and the so-called 'cocktail effect' influence the properties of HEAs [5,7–9], although the influence of these effects are discussed critically in open literature [10–12].

As in any other alloy, however, the properties are predominantly determined by the microstructure. For this reason, a major field of

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research has been dedicated to study microstructure and texture evolution of HEAs during their processing. HEAs have been processed by conventional thermomechanical treatment [13–15], severe plastic deformation [16,17], and additive manufacturing [18,19]. Even as single-phase materials, a variety of mechanical properties have been observed by varying the microstructure especially the grain size and the texture of HEAs [20–24]. In most of these investigations, the Co-Cr-Fe-Mn-Ni system, especially the prototypical equiatomic Cantor alloy (CrMnFeCoNi), has been utilized. This alloy possesses a face-centered cubic (fcc) crystal structure, it has a stacking-fault-energy (SFE) in the range between 18.3 and 27.3 mJ/m^2 [25,26], and exhibits characteristics similar to low SFE metals, such as brass and high-manganese steel [13,25]. Deformation in this and similar HEAs proceeds primarily by dislocation glide. However, at high tensile strains [27], during cryo and room temperature rolling [21], and under severe plastic deformation conditions [16], deformation twinning can be activated as an auxiliary mechanism leading to twinning-induced plasticity (TWIP). Furthermore, by variation of the chemical composition and by lowering the stability of the face-centered cubic (fcc) phase against the hexagonal phase, transformation-induced plasticity (TRIP) was observed in a non-equiatomic $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ alloy [28].

In quaternary and quinary fcc HEAs, weak Brass-type textures have been observed even after 90% rolling at room and cryogenic temperatures [14,29]. Regarding the microstructure, the material has showed slip lines and grain fragmentation at low deformation degrees and deformation-induced twins and shear bands at high degrees. Upon recrystallization annealing, the texture has remained mostly weak but showing the presence of characteristic rolling texture components, such as the {110}<112> Brass, {110}<100> Goss, and {123}<634> S texture components [13]. In turn, the microstructure after recrystallization was found, depending on the annealing temperature, to consist of small equiaxed grains with the presence of recrystallization twins [30]. It is noted that it has been possible to refine substantially the microstructure by the application of conventional heat treatments resulting in an improvement of the mechanical properties [15,22].

Evidently, the key for tailoring the mechanical properties is the understanding of the physical processes of microstructure and texture modification. The purpose of the present study is to investigate the relationship between microstructure and final mechanical properties in the equiatomic Cantor alloy. Therefore, the role of the deformation and recrystallization nucleation mechanisms on the texture and microstructure evolution has been studied. Specific emphasis was placed on deformation and annealing twinning during processing by cold rolling and subsequent heat treatment.

2. Applied methods

2.1. Material and processing

The equiatomic CoCrFeMnNi high-entropy alloy was investigated in this work. The exact chemical composition is given in Table 1. The corresponding SFE of the alloy at room temperature was estimated by Zaddach et al. and Huang et al. to be in the range between 18.3 and 27.3 mJ/m^2 [25,26].

Table 1
Chemical composition of the investigated HEA.

Element	Co	Cr	Fe	Mn	Ni	O (ppm)
(wt.%)	21.8	17.9	20.3	18.8	21.0	135

The alloy was produced as a 500 g-ingot using induction melting in Ar atmosphere followed by hot rolling at 1000 °C and homogenization at 1000 °C for 1 h. The hot-rolled sheet (3.1 mm) was further cold rolled up to 50% thickness reduction (1.55 mm). To investigate the influence of additional heat treatment on the material behavior, the cold-rolled sheets were annealed in the range between 500 °C and 900 °C for 1 h in an air furnace.

2.2. Sample preparation and characterization techniques

Specimens with the dimensions of 12 mm × 10 mm in the rolling (RD) and transverse direction (TD), respectively, were fabricated from the hot-rolled, cold-rolled and annealed sheets using electrical discharge machining. The samples were mechanically ground up to 4000 SiC grit paper followed by mechanical polishing using 3 μm and 1 μm diamond suspension. For X-ray diffraction (XRD) pole figure measurements, the middle layer of the RD-TD section was polished electrolytically at room temperature for 40 s at 24 V, whereas the RD-ND (ND - normal direction) section was electropolished for scanning electron backscatter diffraction (EBSD) using the same parameters as before. The electrolyte used for XRD and EBSD sample preparation consisted of 700 ml ethanol (C_2H_5OH), 100 ml butyl glycol ($C_6H_{14}O_2$), and 78 ml perchloric acid (60%) (ClO_4). Transmission electron microscopy (TEM) samples (~100 μm thick, 3 mm in diameter) were prepared using the same electrolyte as for XRD and EBSD samples in a double jet Tenupol-5 electrolytic polisher with a voltage of 22–24 V and a flow rate of 10 at 4–6 °C.

EBSD analyses were performed in a LEO 1530 field emission gun scanning electron microscope (FEG-SEM) operated at 20 kV accelerating voltage and a working distance of 10 mm. A step size of 100 nm was used for data acquisition. The HKL Channel 5 software was utilized to visualize the ESBD data as well as for data post-processing, removal of wild spikes and non-indexed points, taking at least five neighbor points into account. Subdivision of EBSD mappings into subsets containing only non-recrystallized (non-RX) or recrystallized (RX) grains was performed using an algorithm of the MATLAB®-based toolbox *MTEX* [31–33], as described in Refs. [34,35]. The internal grain/subgrain misorientation was calculated based on the grain reference orientation deviation (GROD-AO, where AO refers to average orientation) value, which takes the average grain/subgrain orientation as a reference. The misorientation threshold value for subdivision was chosen as RX < 1.5° < non-RX. Grains containing less than 10 EBSD data points were disregarded. MTEX was also utilized to calculate the micro-texture orientation distribution functions (ODFs). TEM analyses were performed in a FEI Tecnai F20 TEM operated at 200 kV.

X-ray pole figures were acquired utilizing a Bruker D8 Advance diffractometer, equipped with a HI-STAR area detector, operating at 30 kV and 25 mA, using filtered iron radiation and polycapillary focusing optics. In order to characterize the crystallographic texture, three incomplete (0–85°) pole figures {111}, {200}, and {220} were measured. The macrotexture ODFs were also calculated and visualized using *MTEX*. The volume fractions of the corresponding texture components were calculated using a spread of 15° from their ideal orientation. The overall intensity of the textures was characterized by the respective texture index T which was calculated as [36]:

$$T = \oint [f(g)]^2 dg \quad (1)$$

where $f(g)$ is the orientation density function and g denotes the orientation defined by the three Euler angles $g = (\varphi_1, \Phi, \varphi_2)$.

Mechanical properties were evaluated by uniaxial tensile tests at room temperature and a constant strain rate of 2.5×10^{-3} s^{-1}