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# Mechanical behavior and microstructure of $Ti_{20}Hf_{20}Zr_{20}Ta_{20}Nb_{20}$ high-entropy alloy loaded under quasi-static and dynamic compression conditions

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ABSTRACT

The microstructure and the mechanical behavior of equimolar $Ti_{20}Hf_{20}Zr_{20}Ta_{20}Nb_{20}$ high-entropy alloy in a wide range of initial strain rates between ~$10^{-3}$ $s^{-1}$ and ~$3.4 \times 10^{3}$ $s^{-1}$ were studied. A significant increment in the yield strength with increasing strain rate was observed. The yield strength at ~$3.4 \times 10^{3}$ $s^{-1}$ was about 40% higher than that measured at ~$10^{-3}$ $s^{-1}$. Analysis by electron backscatter diffraction shows that in the low strain rate regime (up to ~10 $s^{-1}$) the deformation occurs mainly in evenly distributed bands, while in the dynamic regime the deformation is strongly localized in macroscopic shear bands accompanied by softening even after the onset of yielding. The Kernel Average Misorientation technique reveals a high level of lattice rotation within these bands that also carries intense shear. In addition, X-ray diffraction line profile analysis indicates that the sharp increase in the flow stress is mostly related to an increase of the dislocation density.



## 1. Introduction

Since the pioneering work by Cantor et al. [1] and Yeh et al. [2], high-entropy alloys (HEAs) have triggered a lot of enthusiasm in materials science community and became a hot topic as indicated by the detailed reviews about this alloy family [3,4]. From the available literature data, two mean families of HEAs have been investigated so far: face-centered cubic (fcc) CoCrCuFeNi-based alloys which transform to body-centered cubic (bcc) structures with addition of Al, Ti, and Mn [5–11], and refractory bcc simple solid solution counterparts initiated by Senkov et al. [12–18] and others [19–25]. Indeed, HEAs have drawn more and more attention due to their outstanding mechanical characteristics besides other attractive properties such as large saturation magnetization [26], high thermal stability and good oxidation resistance. For instance, Gali and George [27] studied the mechanical behavior of CrMnFeCoNi and CrFeCoNi alloys and they found that the strength depends strongly on the temperature between 77 and 1273 K, while a relatively weak strain-rate dependence of the strength was observed in the range of $10^{-3}$–$10^{-1}$ $s^{-1}$. It was also shown that the ductility did not vary inversely with the yield strength. A strong work hardening was observed that was attributed to deformation-induced nanotwinning. Zhu and coworkers [28] studied the nature of incipient plasticity in FeCoCrMnNi HEA by instrumented nanoindentation. The maximum shear stress required to initiate plasticity was found to be within 1/15 to 1/10 of the shear modulus and relatively insensitive to grain orientation. Quasi-static compression tests were performed on an equiatomic CoCrFeMnNi high-entropy alloy in a temperature range between 77 and 1073 K [29]. The underlying deformation mechanisms were determined. These include planar dislocation glide in the normal fcc slip systems in the initial stage of plasticity and enhanced ductility observed at 77 K that was attributed to a dynamical Hall–Petch effect due to deformation twinning. Wu and coworkers have shown that the plastic deformation in multi-component fcc HEAs was caused by the motion of mixed dislocations similar to the conventional fcc metals [30]. It was also found that for annealed FeCoNiCrMn HEAs the hardness versus grain size relationship follows the classical Hall–Petch strengthening, though with a relatively high hardening coefficient [31]. Patriarca and coworkers [32] conducted experimental and theoretical investigations on the plastic behavior of single crystalline FeNiCoCrMn HEA. The critical resolved shear stress obtained experimentally (175 MPa) was in good agreement with the value determined by atomistic-modified Peierls–Nabarro model calculations (178 MPa). At the same time, an abnormally strong temperature dependence of the critical resolved shear stress was reported below 293 K as compared to conventional fcc metals [33]. Stepanov and coworkers [34] have studied the

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microstructure evolution in CoCrFeNiMn HEA during uniaxial compression in the temperature range of 873–1373 K. Discontinuous dynamic recrystallization was found to be active during both warm (below 1073 K) and hot (above 1073 K) deformations. The creep deformation was studied at 880 and 1000 K and a strain rate of $6.7 \times 10^{-6}$ $s^{-1}$ using in-situ neutron diffraction [35]. It was shown that the dominant deformation mode is the dislocation glide at 800 K and diffusion-controlled dislocation creep at 1000 K. Exceptional damage tolerance with tensile strengths above 1 GPa and fracture toughness values exceeding 200 MPa·$m^{1/2}$ in CrMnFeCoNi high-entropy alloy were also reported [36]. Sluggish diffusion kinetics has been often observed as an important effect that contributes to the outstanding properties of HEAs. The diffusion couple method was used to measure the diffusion parameters of Co, Cr, Fe, Mn and Ni in ideal-solution-like Co–Cr–Fe–Mn–Ni alloys [37]. It was shown that the diffusion coefficients in the Co–Cr–Fe–Mn–Ni alloys are indeed lower than those in the reference metals. In addition, the corresponding higher diffusion activation energies of the investigated HEAs accounted for sluggish diffusion in these alloys.

Apart from a recent paper dealing with the dynamic behavior of HEAs by Kumar and co-authors [38] in the vast majority of studies on HEAs, the mechanical properties have been mainly investigated via hardness and compression tests while only a few works have been devoted to the tensile behavior of HEAs [39,40].

However, the application of this new kind of promising materials requires the study of its mechanical performance and microstructure evolution in practical deformation processes such as rolling, machining and impact loading. In these processes the strain rate is in the range between 1 and $10^4$ $s^{-1}$. In this strain rate range, flow stress versus strain rate plot usually displays two regimes according to the active deformation mechanisms: the thermally activated regime at low strain rates and the viscous drag regime at high strain rates. The transition between the two mechanisms was found to occur at the strain rate of about $10^3$ $s^{-1}$ [41,42]. From microstructural point of view, adiabatic effects characterize the dynamic regime: dynamic recrystallization and formation of adiabatic shear bands have been reported [43–46]. However, in some cases mechanical twinning was also observed [47].

In this paper we study the mechanical behavior and the microstructure evolution in equimolar TiHfZrTaNb refractory high-entropy alloy during quasi-static and dynamic compressions. Special emphasis will be placed on the latter loading method.

## 2. Experimental procedures

### 2.1. Sample processing

The alloy of composition $Ti_{20}Zr_{20}Hf_{20}Nb_{20}Ta_{20}$ was obtained according to the following process: master alloys Ti–Zr–Hf and Nb–Ta from the high purity elements (purity exceeding 99.9%) were first arc-melted on a water-cooled copper plate under argon atmosphere. A titanium getter was melted before each alloy fusion in order to capture the residual oxygen in the chamber. Each master alloy was melted twice in order to ensure good homogeneity, and then they were mixed together. Homogeneity in the alloy was obtained by high frequency induction melting in a sectorized cooled copper crucible under helium atmosphere. Finally, the alloy was casted by arc melting. Ingots of about 60 mm in length and 10 mm in diameter were obtained.

### 2.2. Mechanical testing

Uniaxial compression data at strain rates ranging from ~$10^{-3}$ to ~$3.4 \times 10^3$ $s^{-1}$ were obtained at room temperature on specimens 6 mm in diameter and 3.2 mm in height. A conventional testing machine was used up to the strain rate of ~10 $s^{-1}$, while a Direct Impact Hopkinson Pressure Bar (DIHPB) setup was used to generate plastic deformation at higher strain rates up to ~$3.4 \times 10^3$ $s^{-1}$ (initial strain rate) [42]. In the conventional compression tests the following strain rates were applied: $10^{-3}$, $10^{-1}$, 4.53 and 9.3 $s^{-1}$. In the DIHPB technique, originally introduced by Dharan [48], a specimen placed against a Hopkinson pressure bar is impacted by a striker moving at a constant velocity. The striker speed, *Vi*, typically between 5 and 100 m $s^{-1}$, is recorded using two laser beams separated by 18 mm and positioned 30 mm prior impact. Both striker and Hopkinson bar are made of tungsten alloy with 20 mm in diameter, 17.5 g $cm^{-3}$ in density and 1500 MPa in yield strength. In the present study impact velocities of 3.7, 6.14 and 10.34 m $s^{-1}$ were applied in the investigation of the macroscopic response in the dynamic regime.

### 2.3. Electron backscatter diffraction

Samples for electron backscatter diffraction (EBSD) investigations were prepared by mechanical grinding using 1200 to 4000 grit SiC papers followed by a final polishing step using a 20 nm alumina oxide particle suspension (OPS) from Struers™. EBSD investigations were carried out using a Zeiss Supra 40VP FEG scanning electron microscope (SEM). Due to the wide grain size distribution in the as-cast material, a step size between the neighboring measurement positions of 1 μm was used. For statistical purpose, all the scans were performed on an area greater than 1 mm × 1 mm. The data were further processed by OIM™ software version 5 from TexSem Laboratories (TSL). The "grain dilation" procedure with a single iteration step was applied to clean up misindexed points. The average grain size, the fractions of low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs), the image quality (IQ), the grain boundary (GB) and the inverse pole figure (IPF) maps were further extracted from the EBSD scans. In addition, the Kernel Average Misorientation (KAM) function was used to determine local values of intragranular lattice rotation [49]. KAM is numerically defined as the average of misorientation between the pixel of interest and all specified nearest neighbor pixels. For a given point, the average misorientation with all neighbors is calculated with the provision that misorientations exceeding some tolerance value are excluded from averaging. For the present calculation, the 5th nearest neighbor pixels and a maximum misorientation angle of 5° (upper limit for KAM computation) were chosen, giving the largest lattice distortion of 1°/μm at the applied kernel diameter of 10 μm.

### 2.4. X-ray diffraction

The average lattice parameter for the deformed HEA samples was investigated by X-ray diffraction (XRD) using a Philips Xpert Θ–2Θ powder diffractometer operating at 40 kV and 30 mA with CuKα radiation (wavelength: $\lambda = 0.15418$ nm). The scan rate was $1.4 \times 10^{-3}$ degree/s. According to the measured XRD patterns all the studied samples have bcc structure. The average lattice parameter was determined from the diffraction peak positions using the Nelson–Riley method [50].

The lattice defect structure in the samples was studied by X-ray line profile analysis (XLPA). The X-ray line profiles were measured by a high-resolution rotating anode diffractometer (type: RA-MultiMax9, manufacturer: Rigaku) operating at 40 kV and 100 mA with $CuK\alpha_1$ (wavelength, $\lambda = 0.15406$ nm) radiation. The diffraction profiles were evaluated by the Convolutional Multiple Whole Profile (CMWP) method [51]. In this procedure, the diffraction pattern is fitted by the sum of a background spline and the convolution of the instrumental pattern and the theoretical line profiles related to crystallite size and dislocations. The instrumental pattern was measured on a $LaB_6$ standard sample (SRM 660). The area-weighted mean crystallite size ($\langle x \rangle_{area}$) and the dislocation density ($\rho$) were determined by the CMWP method. This evaluation procedure requires the knowledge of the elastic anisotropy factor of the studied HEA crystal which is not available in the literature. Therefore, the elastic anisotropy factor *A* of the present HEA material was estimated from the elastic response of grains with different orientations on nanoindentation. The detail of this procedure is