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# Tension/compression asymmetry in additive manufactured face centered cubic high entropy alloy



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#### ABSTRACT

The high entropy alloy,  $Al_{0.3}$ CoCrFeNi, has been fabricated by direct laser fabrication and its deformation behaviour studied. This alloy exhibited significant tension/compression asymmetry in its work hardening rate and ductility. The exceptionally high work hardening in compression has been attributed to profuse mechanical twinning which has been exacerbated by a strong texture in the as-cast material. The tensile deformed material did not exhibit any mechanical twinning, deformed exclusively by slip, and therefore showed minimal work hardening and poor ductility. The critical resolved shear stress for twinning of the alloy was found to be approximately 240 MPa. © 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

High entropy alloys (HEAs) are a relatively new metal alloy system containing five or more principal alloying elements having a concentration between 5 at.% and 35 at.% [1]. Certain HEAs have been found to form a disordered solid solution structure due to high configurational entropy of mixing. The crystal structure of HEAs can be FCC, BCC, HCP or a combination of these, depending on the concentration of each component element [2] and this alloy class is reported to possess promising physical and mechanical properties [3].

Despite considerable research in to HEAs in the past decade there has been little focus on the deformation behaviour in both tensile and compressive loading. The vast majority of literature report mechanical properties of HEAs in one deformation path (predominately compressive), and only one study was found to contain both tensile and compressive behaviour of a HEA [4]. This alloy (FCC + BCC dual phase Al<sub>0.5</sub>CoCrCuFeNi HEA) exhibited a compressive yield strength of 460 MPa and a strain to failure of 0.52; in contrast the yield strength dropped to 360 MPa, and the elongation to failure dropped by more than half in tension. This paper was the first to reveal that there may be some asymmetry in the deformation behaviour of HEAs. Additional evidence of asymmetry can be found in the literature by comparing the reported tensile and compressive properties of similar alloys by different authors. For example, the medium entropy alloy CoCrFeNi with FCC structure has a tensile yield strength of 200 MPa and ductility of 0.35 in tension [5], but in compression has been reported to have a yield strength of only 135 MPa and strain to failure in excess of 0.75

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http://dx.doi.org/10.1016/j.scriptamat.2016.10.023 1359-6462/© 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved. [6]. A similar discrepancy in the tensile and compressive properties has been reported in other FCC HEAs such as  $Al_{0.1}$ CoCrFeNi [7,8] and CoCrFeMnNi [9,10]. It is not clear if these differences are due to minor variations in the microstructure between the different studies, or if there is a genuine tension/compression asymmetry for this alloy class. We therefore chose here to study the deformation behaviour of the high entropy alloy  $Al_{0.3}$ CoCrFeNi with particular emphasis on differences in its tensile and compressive deformation behaviour. This alloy has a FCC structure and has been noted for its excellent mechanical properties over a wide range of temperatures [11].

Columns (x:y:z – 15:15:100 mm) of the alloy were manufactured by direct laser fabrication (DLF) using a Trumpf Truelaser Cell 7040 using the same deposition parameters provided elsewhere [12]. The composition of the alloy determined by glow discharge optical emission spectroscopy is given in Table 1. Specimens for mechanical testing were prepared by electro-discharge machining, with the mechanical load direction parallel to the build direction, Fig. 1(a). Cylindrical specimens for compression testing had a diameter of 8 mm and a height of 12 mm, and the tensile specimen had a gauge width, gauge length and gauge thickness of 4 mm, 16 mm and 2 mm respectively. Tensile testing was performed using an Instron 100 kN machine and compression testing using a Servotest Thermo Mechanical Test Simulator (TMTS-500 kN). Both orientations were tested at an initial strain rate of  $10^{-3}$  s<sup>-1</sup> and a minimum of three tests were performed in each condition. Microstructural analysis of specimens deformed to different levels of strain was carried out to examine the deformation behaviour with the aid of transmission electron microscopy (TEM) using a Joel 2100F and electron backscatter diffraction (EBSD) in a scanning electron microscope (SEM) Zeiss Supra 55 VP.

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 Table 1

 Bulk chemical composition of direct laser fabricated Al<sub>0.3</sub>CoCrFeNi alloy determined by glow-discharge optical emission spectroscopy.

Alloy	Atomic % (±0.5)				
	Со	Cr	Fe	Ni	Al
Al <sub>0.3</sub> CoCrFeNi	23.4	22.9	23.3	23.1	7.1

The DLF Al<sub>0.3</sub>CoCrFeNi alloy exhibited XRD spectra consistent with a FCC phase (Fig. 1(a)). The grain structure was examined parallel and transverse to the build direction, and representative microstructures are shown in Fig. 1(a). The grain structure is characteristically large and columnar, having an average size of 250  $\mu$ m in the transverse direction and 1250  $\mu$ m in the longitudinal direction. Also shown in Fig. 1 is the crystallographic texture in the build direction, which shows a strong (001) alignment as evident from the EBSD inverse pole figure map (Fig. 1(b)), (100) pole figure (Fig. 1(c)) and strong (200) diffraction peak in the XRD spectra (Fig. 1(a)). This may be attributed to the epitaxial growth of the alloy along the direction of deposition, owing to the rapid cooling rate and steep thermal gradient in the melt pool during laser fabrication as suggested for Ni-base superalloy Rene 41 [13]. As described in [12], DLF Al<sub>0.3</sub>CoCrFeNi alloy contains a small quantity of very fine (approximately 250 nm) Ni- and Al-rich particles at grain boundaries.

Significant differences in mechanical behaviour are illustrated in the flow curves from tension and compression testing (Fig. 2). Although the alloy showed a yield strength of 194 MPa in tension and compression,

the work hardening behaviour after yielding was markedly different in the different loading directions. In compression, the alloy exhibited a substantial and sustained work hardening behaviour without failure for the duration of the test (the test was stopped at true strain of 1.0, Fig. 2(b)). In tension however, the alloy showed limited work hardening and failed at a true strain of 0.38. Tensile failure occurred by the propagation of cracks along the grain boundaries parallel to the tensile axis, Fig. 2(c). This grain boundary cracking resulted in a significant drop in the flow stress. The observed weakness of the grain boundary regions in tension may be due to grain boundary precipitates that develop in this alloy, identified to be enriched in nickel and aluminium with an average size of 250 nm from our previous study [12]. A similar observation was previously made in the literature, where low melting point eutectics formed along columnar grain boundaries and inter-dendritic regions of laser fabricated Rene88DT superalloy, and resulted in the premature failure of the alloy [14]. Flow serrations were evident in the tensile data (inset of Fig. 2a). This may be due to progressive cracking along the grain boundaries during testing. Alternatively, the serration may be due to dynamic strain ageing, which has been observed in several HEAs in certain deformation conditions of temperature and strain rate [11,15,16]. Dynamic strain ageing was previously observed in single crystal Al<sub>0.3</sub>CoCrFeNi at temperatures >873 K and this was correlated with the interaction of solutes, especially aluminium with dislocations [11]. This is consistent with the accepted notion that solute pinning leads to the development of the plastic instabilities responsible for flow serrations [17].





**Fig. 1.** (a) XRD spectra of as-deposited Al<sub>0.3</sub>CoCrFeNi alloy with microstructure longitudinal to direction of deposition (SD) and cross section (CS); (b) Initial orientation of as-deposited alloy using EBSD-IPF map with schematic representation of tensile and compressive specimens with respect to loading directions and (c) (100) pole figure.

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