

Evidence for superplasticity in a CoCrFeNiMn high-entropy alloy processed by high-pressure torsion

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ABSTRACT

A CoCrFeNiMn high-entropy alloy was processed by high-pressure torsion to produce a grain size of ~10 nm and then tested in tension at elevated temperatures from 773 to 1073 K using strain rates in the range from 1.0×10^{-3} to $1.0 \times 10^{-1} \text{ s}^{-1}$. The alloy exhibited excellent ductility at these elevated temperatures including superplastic elongations with a maximum elongation of >600% at a testing temperature of 973 K. It is concluded that the formation of precipitates and the sluggish diffusion in the HEA inhibit grain growth and contribute to a reasonable stability of the fine-grained structure at elevated temperatures. The results show the activation energy for flow matches the anticipated value for grain boundary diffusion in nickel but the strain rate sensitivity is low due to the occurrence of some grain growth at these high testing temperatures.

1. Introduction

High entropy alloys (HEAs) are a new class of materials containing five or more principal elements with a simple crystal structure, such as an *fcc* lattice, which show a potential combination of high solid solution strengthening and good ductility according to its solid solution phase [1,2]. One of the most studied single-phase HEAs is the equiatomic CoCrFeNiMn alloy [3–5]. The high temperature mechanical properties of this alloy were studied earlier but to date the occurrence of superplasticity has not been reported in this HEA [4, 6–9]. It is well established that superplastic flow requires a very small grain size [10] and this may be achieved most readily through the application of severe plastic deformation (SPD) [11,12]. Processing by the SPD technique of high-pressure torsion (HPT) is especially effective in producing ultra-fine grains [13] and this procedure has led to superplastic elongations in numerous metallic alloys [14,15].

There are only a few reports describing the influence of HPT processing on HEAs [16–20] but it was shown for the CoCrFeNiMn alloy that processing by HPT leads to exceptional grain refinement to ~10 nm with a significant strength of ~1.75 GPa, a hardness of ~4.41 GPa and with a very low ductility of ~4% at room temperature. No systematic investigations have been conducted to evaluate the high temperature mechanical behavior of this nanocrystalline CoCrFeNiMn alloy prepared by HPT. Accordingly, the present research was initiated to evaluate the effect of grain refinement due to HPT on the high

temperature mechanical properties of the CoCrFeNiMn alloy. As will be demonstrated, this HEA is capable of exhibiting superplastic elongations of up to >600% when testing in tension at a temperature of 973 K.

2. Experimental material and procedures

An equiatomic CoCrFeNiMn (20 at% each element) alloy was prepared by arc-melting, casting into a bar and then homogenizing at 1273 K for 16 h in an Ar atmosphere: full details of the experimental procedure were given earlier [20]. Polished disks with diameters of 10 mm and thicknesses of ~0.8 mm were prepared from the homogenized alloy and then processed by HPT for 5 turns at room temperature (RT) under an applied pressure of 6.0 GPa at 1 rpm using quasi-constrained conditions in which there is a small outflow of material around the periphery of the disk during the torsional straining [21]. Foils for transmission electron microscopy (TEM) were prepared after HPT processing using a focused ion beam (FIB) Zeiss Nvision 40 FIB facility at 3 mm from the disk centres in the normal sections of the disks so that the normal of the images lay in the shear direction. TEM micrographs were obtained using a JEOL JEM-3010 microscope operating under an accelerating voltage of 300 kV.

Two miniature tensile specimens were cut from symmetric off-centre positions in each disk in order to avoid any inhomogeneities in the central regions of the disks [22]. These specimens were prepared by

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electro-discharge machining with gauge dimensions of $1.1 \times 1.0 \times 0.6 \text{ mm}^3$ and the mechanical properties were examined at temperatures from 773 to 1073 K. The stress-strain curves were recorded at each temperature using initial strain rates from 1.0×10^{-3} to $1.0 \times 10^{-1} \text{ s}^{-1}$ with at least two samples tested under each condition to ensure good reproducibility and with the testing conducted at constant displacement rates. The stress-strain curves were used to determine the ultimate tensile strengths (UTS) and the elongations to failure of each specimen with the elongations carefully checked by measuring the gauge lengths before and after tensile testing. Microstructural characterizations were conducted after testing by examining the gauge lengths and the gripping sections with optical microscopy (OM) and scanning electron microscopy (SEM). For the OM and SEM observations, the samples were ground through 800, 1200 and 4000 grit SiC papers, polished using a 40 nm colloidal silica suspension and then etched with a solution of 50 mL H_2O , 50 mL HCl and 10 g CuSO_4 .

3. Experimental results

A TEM micrograph and a selected area electron diffraction pattern (SAED) of the as-processed CoCrFeNiMn HEA are shown in Fig. 1 at a region $\sim 3 \text{ mm}$ from the disk centre. The image shows that the microstructure of the HEA is highly strained with complicated non-uniform contrast because of the presence of a high density of different types of lattice defects. Many of the grains have irregular shapes with sharp corners and many grains are surrounded by wavy and not well-delineated boundaries. These diffuse boundaries are typical of materials prepared using SPD techniques and they are consistent with the presence of a large volume of high-energy non-equilibrium boundaries [23]. The equiaxed grains visible in Fig. 1 were formed by the fragmentation of elongated grains and it is apparent that the average size of the separate fragments of these structures is $\sim 10 \text{ nm}$. The arrangement of the diffraction spots in semi-continuous circles in the SAED pattern confirms that the microstructure contains boundaries having high angles of misorientation and the appearance of significant streaking of diffraction spots indicates the presence of high internal stresses and elastic distortions of the crystal lattice. It is important to note that the observed diffraction pattern corresponds to the *fcc* phase.

Fig. 2(a) shows the engineering stress-elongation curves after tensile deformation at 773–1073 K with an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$. The results reveal an initial hardening followed by a peak and then gradual softening with the total elongations to failure increasing with increasing temperature up to 973 K and then decreasing. A complete set of curves is shown in Fig. 2(b), (c) and (d) after

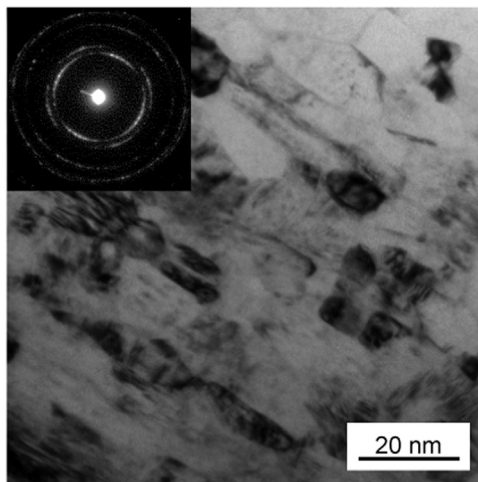


Fig. 1. TEM image and corresponding SAED pattern for the CoCrNiFeMn high-entropy alloy after 5 turns of HPT processing at 1 rpm.

tensile deformation at 873, 973 and 1073 K, respectively, and it is apparent that the total elongation consistently increases with decreasing strain rate. The maximum total elongations recorded in these experiments exceed 600% and this confirms the occurrence of superplasticity which requires an elongation of at least 400% [10]. However, the experimental range of strain rates was not sufficient to reveal the three regions of flow generally associated with conventional superplastic alloys where the measured elongations decrease at both high and low strain rates [24].

Using the data from Fig. 2, Fig. 3(a) shows the flow stress plotted logarithmically against the strain rate where the stress values decrease with increasing temperature and decreasing strain rate. The measured value of the strain rate sensitivity, m , is ~ 0.31 over two orders of magnitude of strain rate.

In order to understand the mechanism controlling the superplastic deformation in the CoCrFeNiMn HEA, the following equation was utilized [25]:

$$\dot{\epsilon} = A \left(\frac{b}{d} \right)^p \left(\frac{\sigma}{G} \right)^n \exp \left(-\frac{Q}{RT} \right) \quad (1)$$

where A is a constant, σ is the flow stress, G is the shear modulus, n is the stress exponent which is equivalent to the reciprocal of the strain rate sensitivity m , b is the Burgers vector, d is the grain size, p is the grain size exponent, Q is the activation energy for deformation, R is the gas constant and T is the testing temperature. Fig. 3(b) shows a semi-logarithmic plot of the strain rate against the inverse of the absolute temperature, T , for an applied stress, σ , of 200 MPa which is represented by the dashed line in Fig. 3(a) and this gives an experimental activation energy, Q , of $\sim 113 \text{ kJ mol}^{-1}$.

Fig. 4 shows the microstructures in the grip and gauge sections of the specimens deformed at 973 K, where the strain rate was $1.0 \times 10^{-3} \text{ s}^{-1}$, the tensile directions are marked as TD and the specimen corresponds to the maximum tensile elongation of $\sim 610\%$. The SEM observations in Fig. 4(b) and (c) were undertaken at positions B and C within the gauge length and D within the gripping area as depicted in Fig. 4(a) where it is apparent that B lies very close to the fracture point of the specimens. The smooth nature of the gauge section in Fig. 4(a), and the lack of any incipient necking, is directly consistent with the requirements for superplastic flow [26].

Fig. 5 shows the microstructures in the grip and gauge sections of the specimens deformed at 1073 K where Fig. 5(a) is an OM image of the fracture tip and the SEM image in (b) and OM images in (c) and (d) correspond to positions B, C and D as illustrated in Fig. 4(a). The strain rate was $1.0 \times 10^{-3} \text{ s}^{-1}$ and the specimen corresponds to the maximum tensile elongation of $\sim 400\%$. The EDS analyses of points marked as A–E are shown in (b).

The possibility of grain boundary sliding (GBS) was examined in these experiments by inspection of the images in Figs. 4 and 5. The images show there is a homogeneous equiaxed microstructure both in the undeformed grip regions and in the deformed gauge sections for the samples pulled at 973 and 1073 K with final grain sizes after deformation of ~ 1.0 and $\sim 5.5 \mu\text{m}$, respectively. Thus, although these specimens failed at high elongations, there is no evidence for any elongated grains within the deformed microstructures and this is consistent with the occurrence of GBS and conventional superplasticity.

A microstructural analysis of the sample tested at 973 K showed clearly the presence of precipitates in the matrix and some of these precipitates are marked by arrows in Fig. 4(b)–(d). It also appears that some precipitates were extracted from the matrix during etching. It was noted earlier that the CoCrFeNiMn alloy has a single-phase *fcc* structure above 873 K but with a mixture of *fcc* and *bcc* phases below 873 K or, under some conditions, with a σ phase which is an intermetallic compound having a tetragonal crystal structure [27]. An earlier study suggested that Cr-rich precipitates (*bcc* and σ phases) are

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