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Effect of a minor titanium addition on the superplastic properties of a CoCrFeNiMn high-entropy alloy processed by high-pressure torsion

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ABSTRACT

A CoCrFeNiMn high-entropy alloy (HEA) with an addition of 2 at% Ti was processed by high-pressure torsion to produce a grain size of ~30 nm and then tested in tension at elevated temperatures from 873 to 1073 K using strain rates from 1.0×10^{-3} to 1.0×10^{-1} s^{-1}. The alloy exhibited excellent ductility at these elevated temperatures including superplastic elongations with a maximum elongation of 830% at a temperature of 973 K. It is shown that the Ti addition contributes to the formation of precipitates and, combined with the sluggish diffusion in the HEA, grain growth is inhibited to provide a reasonable stability in the fine-grained structure at elevated temperatures. By comparison with the conventional CoCrFeNiMn HEA, the results demonstrate that the addition of a minor amount of Ti produces a smaller grain size, a higher volume fraction of precipitates and a significant improvement in the superplastic properties.

1. Introduction

High-entropy alloys (HEAs) have attracted worldwide attention due to their potential beneficial mechanical characteristics such as high strength, good ductility and high thermal stability. HEAs contain five or more elements with each elemental concentration between 5 at% and 35 at% and an intriguing aspect is that their high configurational entropies favor the formation of simple structures based on solid solution phases over the precipitation of brittle intermetallic compounds [1–3]. Generally, a potential combination of high solid solution strengthening and good ductility may be achieved if the solid solution phase possesses a simple crystal structure, such as an *fcc* lattice with a large number of slip systems [4]. An example, and one of the best studied such single-phase HEAs, is the equiatomic CoCrFeNiMn alloy which is a single-phase *fcc* solid solution [5,6]. Experiments show this HEA exhibits very good ductility but its strength is relatively low in the homogenized condition [7]. The strength of the alloy may be increased without significantly sacrificing the ductility by introducing additional strengthening methods such as solid solution or precipitation hardening which require a modification of the chemical composition of the alloy. It is well known in practice that, in addition to the principal elements, HEAs can also contain minor elements with each below 5 at% [8].

The high temperature mechanical properties of HEAs were studied earlier [9–12] and some limited results are now available documenting the occurrence of superplasticity in a number of HEAs [13–17]. A recent brief review summarizes the major superplastic results obtained to date [18] and also includes a description of a recent report of superplastic-like flow in a CoCrFeNiMn HEA [19]. It is well established that superplasticity requires a grain size smaller than ~10 μm [20] and this may be achieved most readily through the application of severe plastic deformation (SPD) [21,22]. Processing by the SPD technique of high-pressure torsion (HPT), where a disk is subjected to a high applied pressure and concurrent torsional straining [23], is especially effective by comparison with other SPD procedures in producing both exceptionally small grain sizes [24–26] and microstructures containing a large fraction of grain boundaries have high angles of misorientation [27]. Thus, processing by HPT appears to be especially attractive for achieving superplastic flow in HEAs. Superplasticity is defined formally as a tensile elongation of at least 400% [28] and there are reports to date of superplastic behavior with a maximum elongation of ~1240% when testing at 1273 K in AlCoCrCuFeNi processed by multiaxial forging [13–16] and a maximum elongation of > 600% at 973 K in CoCrFeNiMn processed by HPT [17]. Superplastic-like flow with a smaller elongation of 320% at 1023 K was also reported in CoCrFeNiMn processed by rolling [19].

Following earlier research on CoCrFeNiMn [17,29], the present research was initiated to provide detailed information on the significance of adding a small amount (2 at%) of Ti to the CoCrFeNiMn

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HEA. A brief study on the $CoCrFeNiMnTi_{0.1}$ HEA reveals that an addition of minor Ti improves the thermal stability of the CoCrFeNiMn HEA by the stability of precipitates [30]. Specifically, the investigation examined the influence of the Ti addition on grain refinement by HPT and on the subsequent high temperature mechanical properties in the HPT-processed condition. As will be demonstrated, the addition of Ti is beneficial and the $CoCrFeNiMnTi_{0.1}$ HEA exhibits enhanced superplastic elongations of up to > 800% when testing in tension at 973 K.

2. Experimental material and procedures

A $CoCrFeNiMnTi_{0.1}$ ($Co_{19.6}Cr_{19.6}Fe_{19.6}Ni_{19.6}Mn_{19.6}Ti_2$ in at%) alloy was prepared using a non-consumable vacuum arc melting technique in a water-cooled copper crucible. After several remeltings to give reasonable homogenization, the ingots were hot forged and then homogenized at 1273 K for 960 min to give an initial grain size of ~200 μm. 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 [31].

Two miniature tensile specimens with gauge dimensions of $1.1 \times 1.0 \times 0.6\ mm^3$ were cut from symmetrical off-centre positions by EDM in each disk in order to avoid any deformation inhomogeneities in the central regions of the disks [32]. The mechanical properties were measured at temperatures from 873 to 1073 K. The stress-strain curves were recorded at each temperature using initial strain rates from 1.0×10^{-3} to $1.0 \times 10^{-1}\ s^{-1}$ at constant displacement rates and with at least two samples tested under each condition to ensure good reproducibility. The stress-strain curves were used to determine the ultimate tensile strength (UTS) and the elongation to failure for each specimen with the elongations checked carefully by measuring the gauge lengths before and after tensile testing. After tensile testing at 973 K the grip sections were polished to a mirror-like quality and hardness measurements were taken using a Vickers microhardness tester with a load of 500 gf and dwell times of 10 s. In practice, these measurements were recorded after the HPT-processed samples were held at 973 K for times in the range of 20–140 min. At every indentation point, the local value of the microhardness, Hv, was determined from the average of five separate hardness values.

The phase constituents were determined using X-ray diffraction (XRD) employing Cu Kα radiation (wavelength λ = 0.154 nm) at 45 kV and a tube current of 200 mA with Rigaku SmartLab equipment. These determinations were conducted in the grip sections after tensile testing at 973 K with initial strain rates of 1.0×10^{-3} to $1.0 \times 10^{-1}\ s^{-1}$ and near the edges of the disks for samples before and after HPT processing. XRD measurements were recorded over a 2θ range from 30° to 100° using a scanning step of 0.01° and a scanning speed of $2°\ min^{-1}$. Microstructural characterizations were conducted after tensile testing by examining the gauge lengths and the gripping sections with scanning electron microscopy (SEM). For the SEM observations, samples were ground through 800, 1200 and 4000 grit SiC papers and then polished using a 40 nm colloidal silica suspension.

3. Experimental results

3.1. Results from tensile testing after HPT

Fig. 1(a) shows a plot of engineering stress against engineering strain after tensile deformation at 873–1073 K with an initial intermediate strain rate of $1.0 \times 10^{-2}\ s^{-1}$. The curves reveal an initial hardening followed by a peak and then gradual softening before failure. The maximum elongation to failure occurs at 973 K and there are lower elongations at both lower and higher temperatures. A set of curves is shown in Fig. 1(b) after tensile deformation at 973 K and it is apparent

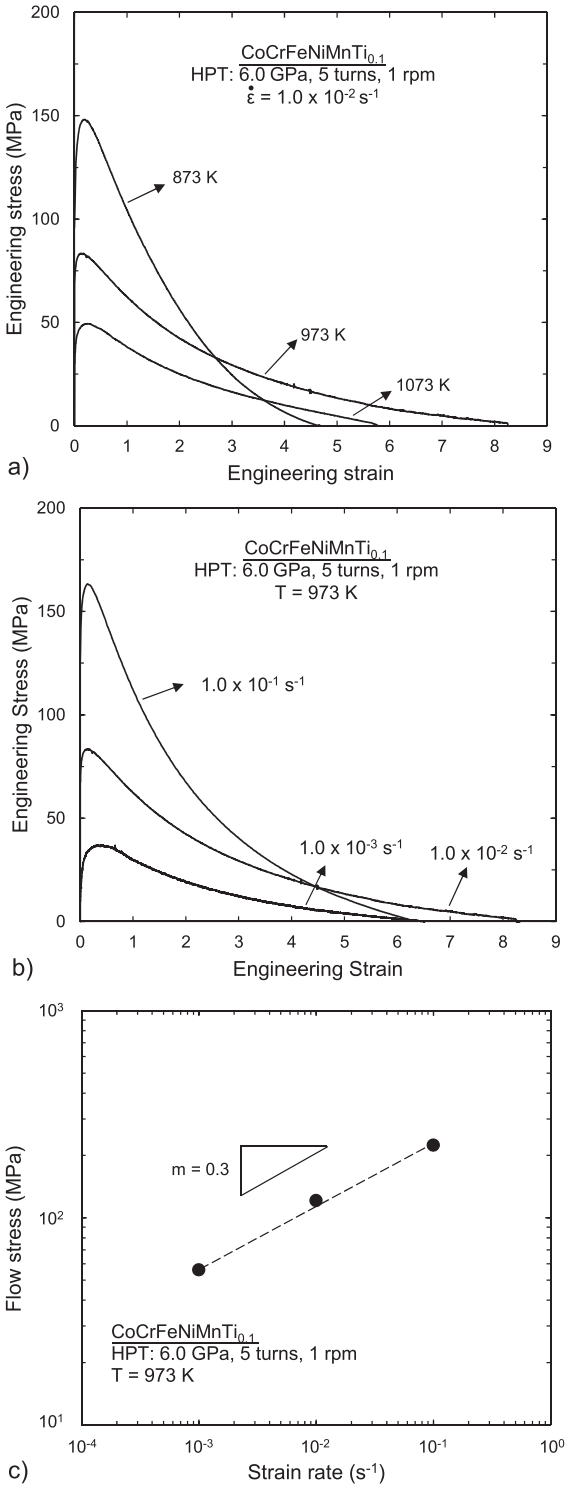

Fig. 1. (a) Engineering stress versus engineering strain for samples processed by HPT through 5 turns at an initial strain rate of $1.0 \times 10^{-2}\ s^{-1}$ and different temperatures, (b) engineering stress versus engineering strain for samples processed at 973 K at different initial strain rates and (c) flow stress versus initial strain rate at 973 K to determine the strain rate sensitivity, *m*.

that the total elongation is a maximum at the intermediate strain rate of $1.0 \times 10^{-2}\ s^{-1}$. This result demonstrates the occurrence of three distinct regions of flow as in conventional superplastic alloys where the measured elongations decrease at both high and low strain rates [33].

The maximum total elongation recorded in these experiments was 830% and this confirms the occurrence of true superplasticity. Using