Materials Science & Engineering A 636 (2015) 188–195

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

High temperature deformation behavior and dynamic recrystallization in CoCrFeNiMn high entropy alloy

N.D. Stepanov [a,*], D.G. Shaysultanov [a], N.Yu. Yurchenko [a], S.V. Zherebtsov [a], A.N. Ladygin [b], G.A. Salishchev [a], M.A. Tikhonovsky [b]

[a] Laboratory of Bulk Nanostructured Materials, Belgorod State University, Belgorod 308015, Russia
[b] National Science Center "Kharkov Institute of Physics and Technology" NAS of Ukraine, Kharkov 61108, Ukraine

ARTICLE INFO

Article history:
Received 25 February 2015
Received in revised form
23 March 2015
Accepted 25 March 2015
Available online 3 April 2015

Keywords:
EBSD
Mechanical characterization
High entropy alloys
Thermomechanical processing
Recrystallization
Shear bands

ABSTRACT

Microstructure evolution in high-entropy alloy CoCrFeNiMn during uniaxial compression to a height reduction of true strain of ≈1.4 in the temperature interval 600–1100 °C was studied. Although some differences was observed in the mechanical behavior of the alloy and the activation energy of deformation in warm (below 800 °C) and hot (above 800 °C) temperature intervals, microstructure evolution at all studied temperatures was found to be accompanied by discontinuous dynamic recrystallization (dDRX). During hot deformation recrystallization was primary associated with nucleation of new grains on the initial grain boundaries, while in the warm interval dDRX was mainly observed in shear bands. The volume fraction of the recrystallized structure was respectively 0.085 and 0.95 at 600 and 1000 °C and the recrystallized grain size was found to be 0.2 and 40.4 μm for 600 and 1100 °C, respectively.

1. Introduction

So-called high entropy alloys (HEAs) are the new class of materials defined as alloys consisting of 5 or more principal elements with nearly equimolar fractions [1–3]. They have received considerable attention in recent years due to their unusual structures and properties. High entropy of mixing associated with multiple principle components is thought to prevent formation of intermetallic phases [1] and promote formation of simple solid solution structures. However, among the equiatomic alloys based on transition metals only a couple have truly single solid solution structures, namely CoCrFeNi and CoCrFeNiMn alloys [4–6]. These alloys consist of single fcc solid solution. Adding other elements like Al, V, Ti, Mo or Nb to CoCrFeNi and CoCrFeNiMn alloys results in formation of ordered or intermetallic phases [7–13]. That is why the CoCrFeNi and CoCrFeNiMn alloys have attracted considerable attention of the researchers.

Remarkable mechanical properties of these alloys were reported in [14–19]. It was demonstrated that they have relatively low yield stress, excellent strain hardening capability and exceptional ductility at ambient and cryogenic temperatures [14,15]. The CoCrFeNiMn alloy also has promising fracture toughness at cryogenic temperature [16]. Much less attention has been paid to mechanical behavior of the CoCrFeNi and CoCrFeNiMn alloys at elevated temperatures [17]. It should be noted that the majority of the published data, especially at elevated temperatures, was obtained during tensile testing [15–17,19]. This deformation scheme limits the maximum attained strain thereby focusing the main attention of the researchers to microstructure evolution associated with low strain. Meanwhile the available data on compressive test is mainly limited to room temperature whereas this method is very useful for investigation of microstructure evolution and mechanical behavior during large deformation in a wide temperature interval.

Although thermo-mechanical treatment via warm or hot deformation is rather rarely applied to HEAs, it can significantly refine the microstructure and enhance the mechanical properties. For example, hot multiaxial forging of the multiphase AlCoCrCuFeNi alloy has enabled formation of structure with the average grain/particle size of 1.5 μm and promote superplastic behavior of the forged alloy at elevated temperature [20–22].

It has been already reported that grain size has a very pronounced effect on mechanical properties of the CoCrFeNiMn alloy [15]. To produce recrystallized grains in the CoCrFeNiMn alloy combination of cold working with subsequent annealing i.e. static recrystallization is usually used [14,15]. Static recrystallization behavior of the alloy has been studied quite thoroughly [23–26]. However, no detailed studies on dynamic recrystallization behavior of the CoCrFeNiMn alloy or any other HEA are available in the literature. It might be supposed that such features as sluggish diffusion [27] or low stacking fault energy [28] could influence on dynamic recrystallization behavior of the

* Corresponding author. Tel.: +7 4722 585416.
E-mail addresses: stepanov@bsu.edu.ru, stepanov.nikita@icloud.com (N.D. Stepanov).

http://dx.doi.org/10.1016/j.msea.2015.03.097
0921-5093/

CoCrFeNiMn alloy. Therefore the aim of the current study is to examine the compressive mechanical behavior and corresponding microstructural response of the CoCrFeNiMn alloy at elevated temperatures in the interval 600–1100 °C.

2. Experimental procedures

The equiatomic alloy with the composition of CoCrFeNiMn was produced by arc melting of the components in high-purity argon inside a water-cooled copper cavity. The purities of the alloying elements were above 99.9(at.)%. To ensure chemical homogeneity, the ingots were flipped over and re-melted a least 5 times. The produced ingots of the CoCrFeNiMn alloy had dimensions of about $6 \times 15 \times 60$ mm^3. Homogenization annealing was carried out at 1000 °C for 24 h in accord with regime used previously for the alloy [9,10,14]. Prior to homogenization, the samples were sealed in vacuumed (10^{-2} Torr) quartz tubes filled with titanium chips to prevent oxidation. After annealing, the tubes were removed from the furnace and the samples were cooled inside the vacuumed tubes down to room temperature due to heat exchange with surrounding air.

Compressive tests were performed on rectangular specimens with dimensions of $7 \times 5 \times 5$ mm^3 using the Instron 300LX machine equipped with radial heating furnace. Testing was performed at temperatures of 600 °C, 700 °C, 800 °C, 900 °C, 1000 °C and 1100 °C. The initial strain rate was of 10^{-3} s^{-1}. To estimate activation energy, additional tests were performed at temperatures of 700–1100 °C and strain rates of 10^{-2} s^{-1} and 10^{-4} s^{-1}. All samples were compressed to height reduction of 75% corresponding to true strain of ≈ 1.4. In addition, specimens were compressed to height reductions of 25% and 50–60% (true strain of ≈ 0.3 and ≈ 0.7–0.9) at temperatures of 700 °C and 1000 °C and strain rate of 10^{-3} s^{-1}.

The microstructure of compressed specimens was examined on the plane parallel to the compression axis on the mid-thickness of the specimen. Electron backscattered diffraction (EBSD) was mainly employed for microstructure characterization. A FEI Nova-NanoSEM 450 scanning electron microscope (SEM) equipped with a Hikari EBSD detector was used to produce EBSD maps. Samples were carefully mechanically polished and then underwent electropolishing in mixture of 90% acetic and 10% perchloric acids at room temperature and applied voltage of 27 V for 15 s. TSL OIM Analysis 6 was used to process EBSD data and generate inverse pole figure (IPF) maps. On the presented maps high angle boundaries (HABs) are indicated with black lines and low angle boundaries (LABs) are indicated with white lines. Points having low confidence index (CI ≤ 0.1) were excluded from analysis and are depicted as black dots in presented IPF maps.

3. Results

3.1. Mechanical behavior

True stress–true strain curves obtained during compressive testing of the CoCrFeNiMn alloy are shown in Fig. 1. In addition values of some important characteristics (yield strength $\sigma_{0.2}$, and steady state flow stress σ_{ss}) are summarized in Table 1.

It should be noted that all samples have been compressed to the maximum height reduction ($\approx 75\%$, corresponding to true strain of ≈ 1.4) without any signs of fracture. The mechanical behavior of the alloy significantly depended on deformation temperature. In the interval 900–1100 °C the alloy demonstrated deformation curves with a well-defined steady state flow stage following a short hardening stage in the very beginning of deformation. In contrast the alloy compressed in the interval 600–800 °C continuously

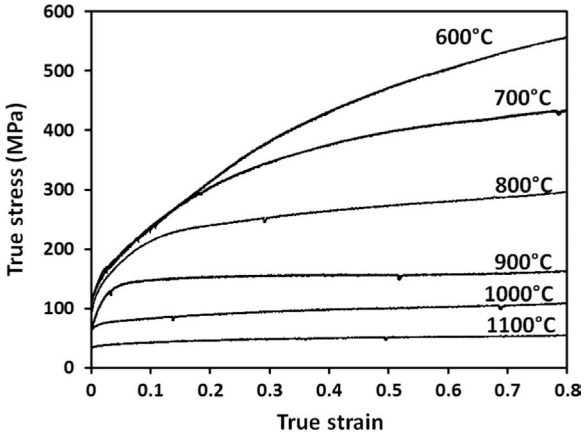

Fig. 1. Stress–strain curves obtained during compressive testing of the CoCrFeNiMn alloy at strain rate of 10^{-3} s^{-1} and temperatures of 600–1100 °C.

Table 1
Compressive mechanical properties of the CoCrFeNiMn high entropy alloy at strain rate of 10^{-3} s^{-1} and temperatures of 600–1100 °C.

Temperature (°C)	$\sigma_{0.2}$ (MPa)	σ_{ss} (MPa)
600	125	–
700	100	–
800	87	–
900	68	156
1000	66	100
1100	36	50

strengthened during deformation till the maximum strain. Strain hardening capability increases with decreasing deformation temperature. Serrations are observed at two minimum temperatures of deformation; the number of serrations decreases with temperature increasing.

To establish whether the difference between the deformation behavior in two temperature intervals is associated with a change in the mechanism of microstructure evolution, activation energy of deformation for these temperatures was determined. To this end compression tests at strain rates of 10^{-4} s^{-1} and 10^{-2} s^{-1} in the interval 700–1100 °C were performed. The deformation behavior of the alloy under these conditions was found to be followed the above described trend, i.e. decrease in strain rate and increase in temperature decreased propensity to strain hardening to almost zero at 1100 °C and 10^{-4} s^{-1}. The values of yield strength $\sigma_{0.2}$ and steady state flow stress σ_{ss} obtained for strain rates of 10^{-4}–10^{-2} s^{-1} and temperatures 700–1100 °C are summarized in Table 2. For low temperatures (700 and 800 °C) flow stresses at $e=0.4$ were taken as σ_{ss}.

The relation between strain rate and temperature during high-temperature deformation can generally be described with the well-known Zener–Hollomon parameter $Z(\dot{\varepsilon})$ [29–31]:

$$Z(\dot{\varepsilon}) = A\dot{\varepsilon}^n = \dot{\varepsilon}\exp(Q/RT), \tag{1}$$

where Q is the activation energy, n is the stress exponent, A is a constant sensitive to the deformation mechanism, and R is the gas constant. The parameters n and Q can be determined as

$$n = \left.\frac{\partial \ln \dot{\varepsilon}}{\partial \ln(\sigma_{ss}/G)}\right|_T. \tag{2}$$

$$Q = -R\left.\frac{\partial \ln \dot{\varepsilon}}{\partial(1/T)}\right|_\sigma \equiv nR\left.\frac{\partial \ln(\sigma_{ss}/G)}{\partial(1/T)}\right|_{\dot{\varepsilon}}, \tag{3}$$

where $\dot{\varepsilon}$ is the deformation strain rate, σ_{ss} denotes the steady state flow stress, n the stress exponent, R the gas constant, and G is the shear modulus of the CoCrFeNiMn alloy at given temperature,