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Elastic and plastic deformations in a high entropy alloy investigated using a nanoindentation method

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ABSTRACT

We employed instrumented indentation technique to study the elastic and plastic deformations in a face-centered cubic (fcc) high-entropy alloy NiFeCoCrMn. Single-crystal Ni was also examined for direct comparison. Tests were carried out using indenters with different tip radii to investigate the effect of indented volume on deformation processes. It was found that when the tip radius increased, the shear stress required for the occurrence of indentation pop-in decreased, which was attributable to a higher probability to find dislocations under a larger tip radius. We proposed a statistical model to describe the results quantitatively. In the plastic region, NiFeCoCrMn was much stronger than Ni, presumably resulted from a large lattice distortion in the multicomponent NiFeCoCrMn alloy. We found that the response of both materials at large indentation depth could be described by the classical Nix-Gao model, but when the indentation depth was shallow, the indenter tip must be treated as a sphere and Swadener's model offered a better description. In any event, it was necessary to introduce a scaling factor *f* to describe the effective stressed volume underneath the indenter tip to compensate for the overestimated hardness values. A map of indentation pressure against dislocation density was also summarized in this study.

1. Introduction

High entropy alloys (HEAs) have received large attention from the materials science community due to their extraordinary properties [1] since they were introduced about a decade ago [2]. HEAs are multicomponent alloys containing several components (often 5) in equal atomic proportions. This new class of alloys is, in principle, expected to exhibit a large degree of mutual solubility in a single-phase structure, instead of complex ordered intermetallics, as a result of having high configurational entropy. However, the majority of HEAs is actually multiphase [3–12], suggesting a high entropy, sometimes, is not a guarantee for the formation of simple solid-solution phase, and the competition between entropy and enthalpy eventually determines the final phase formation.

There are, in fact, only a few HEAs with a true single-phase face-centered cubic (fcc) [13,14], body-centered cubic (bcc) [15–18] or hexagonal close-packed (hcp) [19] structure, in which the high-entropy effect dominates. Among them, the equiatomic fcc-NiFeCoCrMn alloy [13] is a prominent example that exhibits high phase stability, and its mechanical properties and microstructure have been studied [20–26]. For instance, Otto et al. [23] reported that both yield strength, σ_y, and tensile strength, σ_{UTS}, of this HEA increased with decreasing temperature, and the temperature dependence of σ_{UTS} was determined by deformation twins at low temperature, but the temperature dependence of σ_y was from a solid-solution effect. Wu et al. [27,28] further investigated the effect of temperature on the mechanical strength of this fcc-NiFeCoCrMn system, and found that each constituent elements produced different strengthening effects on the alloys, and the effect of temperature depends on the number and the types of constituents.

Recently, studies on the elastic–plastic deformation behavior of HEAs using micromechanical tests, e.g., micropillar compression [15] and nanoindentation [26], also began to emerge. These micromechanical tests have major advantage of revealing the intrinsic properties of a material with a minimum possible influence from 2-dimensional microstructural defects (e.g., grain boundary, twin boundary). Zou et al. [15] conducted compression on high-entropy alloy NbMoTaW micropillars and observed a higher compression strength in the alloy pillar as compared to pure Nb, Mo, Ta, and W pillars, indicating homogeneous intermixing of constituent elements can produce strong lattice friction in the multicomponent

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HEA. They also observed the strength of bcc HEA micro-pillar is about 3 times higher than that of their bulk counterparts, suggesting some kind of sample size effect.

The elastic–plastic deformation behavior of HEAs has also been characterized by nanoindentation technique. For example, Zhu et al. [26] studied indentation pop-ins and determined the critical shear stress for the incipient plasticity in the fcc-NiFeCoCrMn alloy. Their results indicated that plastic yielding of the NiFeCoCrMn alloy is different from that of the pure metals. Other papers [29,30] also reported that yield strength of pure metals (e.g., Mo, Ni, Cr) depends on the tip radius of the indenter, and specifically the smaller is the tip radius, the higher is the yield strength.

In this study, the elastic/plastic deformation of a high entropy alloy NiFeCoCrMn is studied using instrumented indentation technique and directly compared with that of a nickel single crystal. The indented volume was regulated by the applied load and the radius of the indenter tip, and its effect on the deformation behavior of the HEA was evaluated.

2. Materials and methods

Samples with the nominal composition of NiFeCoCrMn (in atomic proportion) were prepared by arc-melting a mixture of the constituent elements (purity > 99 wt%) in a Ti-gettered high-purity argon atmosphere. The alloy ingots were remelted four times in high-purity argon atmosphere to ensure their homogeneity. In the last melting, the liquid melt was suction-cast into a water-cooled rectangular Cu mold with a dimension of 30 mm W × 60 mm L × 3 mm T. The as-cast ingots were homogenized at 1200 °C for 4 h, and then cold rolled to 50% reduction in thickness. Rectangular samples were sliced from the rolled plates, ground, and polished to a mirror finish. The polished samples were additionally annealed at 1000 °C in vacuum for 2 h to induce recrystallization and grain growth, and to remove residual surface stresses from the mechanical polishing. The microstructure of the annealed alloy was examined using optical microscopy and SEM, and the grain size was found in the range of 60–120 μm. XRD pattern has shown a single phase fcc-NiFeCoCrMn alloy with the lattice parameter of about 3.594 Å. For comparison, a nickel single crystal was also tested. The single-crystal Ni(100) sample was prepared using a directional floating zone melting technique [31], and the test sample was ground and electropolished in an electrolyte containing 40% sulfuric acid before the indentation tests.

Instrumented indentation tests were performed on samples at room temperature using a Hysitron Triboindenter (Hysitron Inc., Minneapolis, MN). Three diamond indenters (two Berkovich tips and a cube corner tip) were used to evaluate the elastic–plastic response, and the tip radii of these three tips were calibrated on single-crystal tungsten to be 80 nm (cube corner), 255 nm (Berkovich) and 759 nm (Berkovich). To avoid the overlap of the plastic zone underneath the indenter, indentation tests were conducted with a 6 μm interval with a maximum load of 200 μN for tips R = 80, 255 nm and 1000 μN for the tip R = 759 nm. At least 70 indents were made for each tip radius. During each indentation test, a constant loading rate of 400 μN/s was imposed to eliminate loading rate effect, and the maximum load was held for 1 s before unloading. Indented sites are within the same grain and at least 6 μm away from the grain boundary to avoid possible effects of orientation and grain boundary. To investigate the plastic behavior, indentation tests were also performed in a neighboring grain with a maximum load of 10 mN and a 20 μm interval. A Berkovich indenter (R = 255 nm) was used, and its area function was calibrated with fused silica.

An atomic force microscope MFP-3D™ (Asylum Research Inc, Santa Barbara, CA) was utilized under the air image mode to evaluate the sample surface before and after indentation tests. The probe (AC160TS-R3) used for imaging has a tip radius of about 9 ± 2 nm. Both the annealed NiFeCoCrMn and single-crystal Ni have an rms roughness value of about 2 nm.

3. Results

3.1. Elastic response of NiFeCoCrMn

In an indentation test, the transition from elasticity to plasticity is marked by the first pop-in on the load–displacement curves, as shown in Fig. 1a and b, for NiFeCoCrMn and single-crystal Ni, respectively. In the figures, three different tip radii are used and it is evident that the larger is the tip radius, the higher is the load to reach a given penetration depth for the occurrence of pop-in. The residual indented marks from the indenters (R = 80 and 759 nm) were examined by AFM, as shown in Fig. 2. It is noted that the pyramidal indenter actually exhibits a spherical bottom with a curvature that is consistent with the calibrated tip radii.

The elastic part of the loading curve prior to a pop-in event is described by the Hertzian relation,

$$P = \frac{4}{3}E_r\sqrt{Rh^3} \tag{1}$$

where R is the tip radius of the indenter, h is the indentation depth, $E_r = [(1-\nu_{in}^2)/E_{in}+(1-\nu_s^2)/E_s]^{-1}$ is the reduced elastic modulus of the tested sample, (ν_{in}, E_{in}) and (ν_s, E_s) are the Poisson's ratio and elastic modulus of the indenter and sample, respectively. Data of P-$h^{1.5}$ pairs at pop-ins obtained from NiFeCoCrMn and Ni single crystal under the indenter (R = 255 nm) are plotted in Fig. 1c and d. The reduced elastic moduli for NiFeCoCrMn and Ni are calculated to be 190.21 ± 1.22 and 187.18 ± 1.59 GPa, respectively. Using these E_r values, the elastic loading curves for other tip radii R = 80 and 759 nm are found to agree with the Hertzian relation. Using the mechanical parameters for the diamond indenter (ν_{in} = 0.07, E_{in} = 1141 GPa) [32] and samples ($\nu_{HEA} = 0.26$, $\nu_{Ni} = 0.31$) [28], the elastic moduli of NiFeCoCrMn and Ni are deduced as 212.62 and 202.2 GPa, respectively. The fact that the NiFeCoCrMn alloy has a higher elastic modulus but a larger lattice parameter than Ni (0.359 nm for NiFeCoCrMn and 0.352 nm for Ni) suggests a stronger bond in the alloy.

3.2. Plastic response of NiFeCoCrMn

Load-displacement curves from indentation tests of NiFeCoCrMn and single crystal Ni at a high load of 10 mN are shown in Fig. 3a. A shallower indentation depth indicates the HEA is harder than Ni. In Fig. 3b, hardness values of these two materials are plotted against the indentation depth, h. Both curves exhibit similar trend and the HEA is harder than pure Ni, i.e.,

$$H_{HEA} = H_{Ni} + \Delta H \tag{2}$$

Hardness is also noted to decrease with increasing h, revealing an indentation size effect (ISE) in both materials. Similar observation has been made in several crystalline materials [33,34], and Nix and Gao [35] proposed the dependence of the hardness H on depth h as:

$$H^2 = H_0^2(1 + h^*/h) \tag{3}$$

where H_o corresponds to the hardness at infinite depth, and h^* is the characteristic length that depends on the indenter shape. The linear relation between H^2 and $1/h$ appears to apply well in the micro-indentation scale, whilst significant deviation from the