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Tensile properties of high- and medium-entropy alloys

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ABSTRACT

Equiatomic, face-centered-cubic, high- and medium-entropy alloys were arc melted, hot-rolled to produce recrystallized sheets, and tensile tested. The alloys having the compositions CrMnFeCoNi and CrFeCoNi exhibited a strong temperature-dependent decrease in strength with increasing temperature from −196 °C to 1000 °C, and a relatively weak strain-rate dependence (at 10^{-3} and 10^{-1} s^{-1}). Ductility did not vary inversely with yield strength; rather, when strength doubled as the test temperature was decreased from room temperature to −196 °C, elongation to fracture increased by a factor of 1.5 to >60%. A high degree of work hardening, possibly due to deformation-induced nanotwinning, postpones the onset of necking and may be the reason for the ductility increase.

1. Introduction

Recently, there has been considerable interest in multi-element, equiatomic alloys and their derivatives e.g.[1–20], that are often referred to in the literature as high-entropy alloys. In ideal solid solutions, the configurational entropy, $\Delta S_{conf} = -R\Sigma n_i ln(n_i)$, where n_i is the atomic fraction of the ith element and R is the gas constant, increases with the number of alloying elements. It has been hypothesized that, when the number of alloying elements increases beyond five, the contribution of configurational entropy to the total free energy becomes significant enough that it can overcome the enthalpies of compound formation and phase separation, thereby stabilizing the solid solution state relative to multi-phase microstructures [1]. Based on this logic, a high-entropy alloy was defined as one with at least five major elements whose individual atomic concentrations are between 5 and 35% [1,7]. Alloys based on one principal metallic element were classified as low-entropy alloys and those comprised of two to four principal elements as medium-entropy alloys [7]. In all such multi-element alloys, the configurational entropy is obviously maximized when the alloying elements are present in equal atomic concentrations [1]; consequently, much of the high-entropy alloy research has tended to focus on alloys consisting of multiple principal elements in roughly equal proportions.

While the above concepts can, in principle, explain why *some* multi-element alloys are single-phase solid solutions [2,13,14,16,17,19,20], most of the so-called high-entropy alloys reported in the literature contain multiple phases [e.g.,4-12,15]. The reason for this is that configurational entropy cannot usually overcome the other driving forces of phase stability, such as enthalpy and non-configurational entropy, which often have stronger influences [19]. Additionally, in alloys consisting of multiple phases, the configurational entropy is actually lower than that implied by the equation above for a single-phase ideal solid solution [19]. Therefore, to study the mechanical properties of *high-entropy* alloys, it is important that truly single-phase solid solution alloys be investigated.

Here we investigate the equiatomic, FCC-structured quinary alloy (CrMnFeCoNi), first reported by Cantor et al. [2] and later confirmed to consist of a single solid-solution phase [19,20]. For comparison with this high-entropy alloy, we investigated a medium-entropy quaternary alloy (CrFeCoNi) that is comprised of the same elements as the Cantor alloy minus the Mn; this quaternary alloy is also single-phase FCC [17]. The tensile properties of neither of these alloys have so far been published. We report here our initial findings on the microstructure and tensile behavior of these alloys, which were produced by arc melting, drop casting and hot rolling, as described below.

2. Materials and methods

Starting from the pure (>99.9% purity) constituent elements in bulk form, two alloys having the equiatomic compositions CrMnFeCoNi (HE-1) and CrFeCoNi (HE-4) were produced by arc

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melting under argon atmosphere. The arc-melted buttons were flipped and remelted five times to promote thorough mixing and to allow the regions in contact with the water-cooled copper hearth to become fully incorporated in the melt. After the fifth remelt, the arc-melted buttons were drop cast into a rectangular copper mold measuring 127 mm × 25.4 mm × 12.7 mm. Weight losses after melting and casting were ∼1% and <0.1% for the HE-1 and HE-4 alloys, respectively. Since there was negligible weight loss in the alloy without Mn, it was assumed that the entire weight loss in HE-1 was due to evaporation of Mn (consistent with its much higher vapor pressure). To compensate for this weight loss, we added an extra gram of Mn per 100 g of raw materials.

The drop-cast bars exhibited dendritic segregation; therefore, they were first annealed at 1000 °C for 24 h in vacuum to homogenize their compositions. After homogenization, the alloys were clad with stainless steel sheet and hot rolled to break down their cast structures and obtain a recrystallized microstructure. The purpose of the cladding was to minimize heat loss to the unheated rolls during the rolling operation. Before the first rolling pass, the clad bars were pre-heated at 1000 °C for 1 h; between all subsequent passes, reheating to 1000 °C was done for ∼15 min. For the first five rolling passes, a 10% reduction in thickness per pass was used; after that, a 20% reduction in thickness per pass was used for all subsequent passes until a final sheet thickness of ∼1 mm was reached. Therefore, starting from the initial thickness of the drop-cast bar (12.7 mm), the total reduction in thickness after rolling was ∼92%.

To evaluate the microstructures of the alloys, transverse sections were cut by electric discharge machining (EDM) from the homogenized bars and hot-rolled sheets. The sections were mounted in epoxy, ground through 600 grit SiC paper, and polished using Vibromet machines, first with 0.3 μm Al_2O_3 and then in a suspension of colloidal silica having a particle size of 60 nm. The polished samples were examined in an XL30 FEG scanning electron microscope (SEM) in the backscattered electron mode. Specimens from the homogenized bars were also used to (a) obtain x-ray diffraction patterns in a Scintag diffractometer and (b) determine their melting points in a NETZSCH 404 C differential scanning calorimeter.

From the rolled sheets, dog-bone shaped specimens with gage sections of 10 mm × 2.5 mm × 0.63 mm were cut by EDM with their long axes parallel to the rolling direction. They were subsequently ground through 1200 grit SiC paper, and stress relieved at 900 °C for 1 h in vacuum. Tensile tests were performed on a screw-driven Instron machine at engineering strain rates of approximately 10^{-3} and 10^{-1} s^{-1} at temperatures from −196 °C to 1000 °C. The cryogenic tests were performed with the specimens completely immersed in liquid nitrogen; tests at room temperature and higher were performed in vacuum. Prior to testing, seven, roughly equally spaced Vickers indents were made on the gage sections using a micro-hardness tester and a 300-g load. The distance between adjacent indents was measured before and after the tensile tests and averaged for each specimen to get the uniform elongation to fracture (the two indents on either side of the fracture plane were excluded from the measurements). Occasionally, all the indents could not be located after testing; in those cases, the total change in specimen length divided by the gage length was used as a measure of ductility (implicitly assuming that all the deformation occurred in the gage section). Fracture surfaces of the tensile tested specimens were examined either in an XL30 or Hitachi S4800 FEG scanning electron microscope.

3. Results and discussion

Backscattered electron images of the cast and homogenized microstructures are shown in Fig. 1 (a) and (b). Both alloys are essentially single-phase; however, HE-1 contains isolated dark particles that are not present in the HE-4 alloy. Energy dispersive X-ray spectroscopy was performed on these particles. Oxygen, chromium and manganese peaks were detected, which leads to the conclusion that the particles are oxides. Since they were absent in the Mn-free HE-4 alloy, the oxygen contamination is likely associated with the Mn, but whether it was introduced through the raw

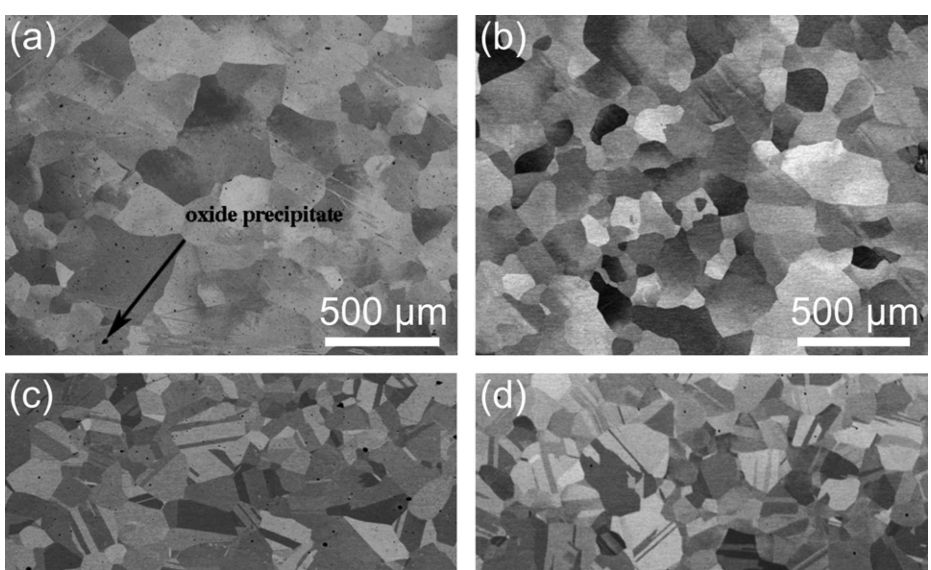

Fig. 1. Backscattered electron images of (a) CrMnFeCoNi (HE-1), (b) CrFeCoNi (HE-4) after casting and homogenization, and (c) CrMnFeCoNi (HE-1), (d) CrFeCoNi (HE-4) after hot rolling.