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Effects of ultrasonic surface mechanical attrition treatment on microstructures and mechanical properties of high entropy alloys

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ABSTRACT

Most high entropy alloys (HEAs) are cast to form single phase solid solution. Their hardness and strength at room temperature under the as-cast condition are typically lower than expectation. In the research, the ultrasonic surface mechanical attrition treatment (SMAT) is conducted on the surface of two HEAs, FeCoNiCrMn and FeCoNiCrMn-Al, to upgrade their room temperature surface characteristics. By proper SMAT multiple paths, the grain size can be reduced from ~50 μm down to ~0.1–1 μm, the hardness increased from ~2.5–5.0 GPa up to ~5.0–8.5 GPa, and the tensile strength and elongation can be nearly doubled. The gradient refined and strengthened surface layers are demonstrated to appreciably upgrade the HEA performance. The strengthening mechanisms and superposition rules are established and are compared well with the experimental measurements.

1. Introduction

Recently, multiple elemental high entropy alloys (HEAs) [1–3], either of the single phase face-centered cubic (FCC) solid solution or of the dual FCC and body-centered cubic (BCC) phases mixture, have attracted attention in terms of their alloy-composition optimization, mechanical-property exploration, and related physical and chemical characteristics [4–9]. While the high temperature heat resistance and sluggish diffusion have opened the window for various HEAs for creep and radiation resistance applications, the room temperature hardness and yield strength, especially for the single FCC phase solid solution, are often lower than expectation, as compared with Ni or Fe based superalloys [10–12].

A newly developed surface nano-crystallization process, surface mechanical attrition treatment (SMAT) [13,14], is modified from shot peening, which uses much large size of balls (1–10 mm), higher frequencies (10–100 kHz), and slower impact velocities (1–20 m/s) [15,16]. Comparing with shot peening, the impact directions of the balls onto the sample surface are rather random due to the random flying directions of the balls inside the vibration chamber. Each impact will induce plastic deformation with a high strain rate in the surface layer of the sample, as schematically shown in Fig. 1. Conventional shot peening is a directional process in which the angle between the shot jet and the sample surface is normally fixed, close to 90° in many cases. But in the SMAT, random directional impacts of the balls onto the sample are needed in order to facilitate the grain refinement process.

It is well known that many material failures are related to the surface conditions, so a proper surface modification may improve the life span and behavior. SMAT is a decent way to introduce a gradient grain refinement and nano-crystallization near the surface. Meanwhile, the properties of the bulk materials can be preserved because of the undeformed matrix inside. Compared with conventional surface nano-crystallization coating and electrodeposition such as physical vapor deposition (PVD) or chemical vapor deposition (CVD), which may be concerned about the chemical composition of the film and the quality of the bonding between the coated nanostructured layer and substrate, SMAT is the advanced way to retain the overall phases and composition [17], and the gradient SMAT-affected zone can be extended to several hundreds of micrometers in depth.

Previous efforts have been made to explore the SMAT effects on various pure metals and alloys, such as Fe, Cu, 304 stainless steel, etc. [18–23]. In this research, we adopt the SMAT as surface processing for two common HEAs, namely, the FeCoNiCrMn (single FCC phase solid solution) and FeCoNiCrMn-Al (a mixture of dual FCC and BCC phases). By multiple SMATs paths, the resulting microstructure and mechanical properties are systematically examined and discussed. The

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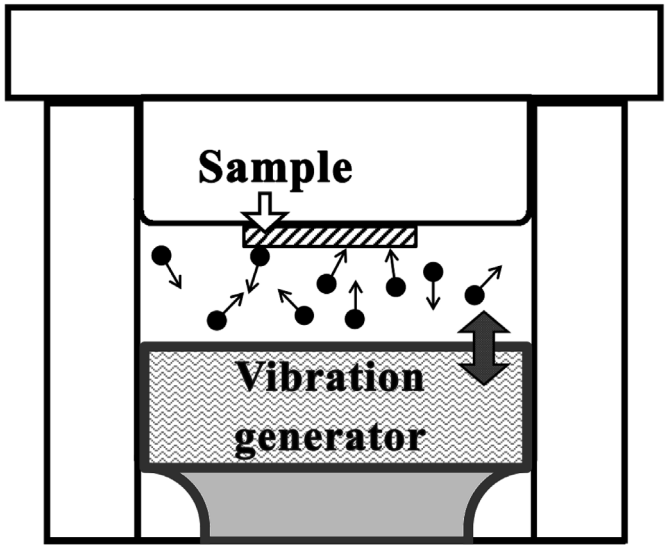

Fig. 1. Schematic illustration of the SMAT set up.

strengthening mechanisms are also explorted.

2. Experimental procedures

In this study, two high entropy alloys are adopted as the tested materials, namely, $Fe_{20}Co_{20}Ni_{20}Cr_{20}Mn_{20}$ (in at%) with single FCC phase and $Fe_{18}Co_{18}Ni_{18}Cr_{18}Mn_{18}Al_{10}$ (in at%) with dual FCC and BCC phases. These two alloys were cast by drop casting system with induction furnace in an atmosphere of high-purity argon and the ingots were re-melted three times to ensure chemical homogenization. The starting pure elements are all in high purity greater than 99.9 wt%. Table 1 summarizes the resulting compositions measured form the two as-cast alloys.

The SMAT set-up in Fig. 1 contains a cylindrical chamber measuring 70 mm in diameter and 20 mm in height. A plate measuring 40 × 20 × 2 mm was set on the top of the SMAT chamber. The plate sample is fixed tightly on the top holder by four screws in avoiding loosening during SMAT. The SUJ2 bearing steel balls with smooth surface and high hardness in the R_C scale of 62 are applied as the energy deliverer and are placed in a reflecting chamber that is vibrated by a vibration generator with a fixed vibration frequency ν = 20 kHz. The vibration amplitude, A, was chosen to 60 μm. The size of the ball was selected the 2 mm in diameter. The density of the ball of size, D, is fixed to be 7.8 g/cm^3. In order to maintain the fixed ball coverage area of 25% inside the chamber, the 2 mm ball case would install 10 g of the total ball weight. Throughout the SMAT experiment, the SMAT time duration is set to be 15 min for each specimen.

The analysis of X-ray diffraction (XRD) was performed by using the SIEMENS D5000 X-ray diffractometer, with Cu K_α radiation λ = 1.5406 Å. The working voltage and current were set to be 40 keV and 30 mA. The SMAT samples with ultra-fine grain microstructure were characterized by scanning electron microscopy (SEM), using the etching reagent of acetic acid + perchloric acid + ethanol (with the volume ratio of 16:4:5). The orientation tests were conducted by using a Gatan Alto 2500 Cryotransfer system interfaced to a field emission gun Zeiss Supra 55 SEM with an EBSD system. To enhance the pattern quality, the specimens were further electropolished with an electrolyte, prior to the establishment of Kikuchi patterns by EBSD. Crystal information is obtained from the EDAX Genesis analytical system computer software.

The morphology of grains/subgrains and dispersion of reinforcement phase in matrix and interface between the reinforcement and matrix were examined by Tecnai G20 field emission transmission electron microscopy with an operating voltage of 200 kV. The cross-sectional transmission electron microscopy (TEM) foils of the SMAT samples were fabricated using the dual-3beam focused-ion-beam (FIB) system (Seiko, SMI3050) with an operating voltage of 30 kV and an ion beam current of 1 pA. In order to observe the nanocrystal structure near the treated surface, the TEM samples of the HEAs were prepared in the dual FIB using a trenching and liftout technique.

The Vickers hardness (Hv) was measured by the SHIMADZU HMV-2 system and with a load of 500 g for 15 s. The hardness of the SMAT specimens was measured from the cross-sectional surface that each datum was tested with an interval of 5 μm by the MTS Nano Indenter XP System. The tests were operated with the displacement rate about 10 nm/s under the continuous stiffness measurement (CSM) mode, and the allowable vibration drift of the environment were controlled under 0.05 nm/s. The indented depth limit was set to be 1200 nm. The as-cast and SMATed samples, which were bombarded at the both sides for the same time period, were then processed into tensile test specimens. The tensile tests using the reduced dog-bone tensile specimens in accordance with the E8M standard, with a gauge length of 12 mm, gauge width of 3 mm and gauge thickness of 2 mm, were performed using the Instron machine, deformed at the initial strain rate of 1×10^{-3} s^{-1}, well within the quasi-steady state.

Table 1
The SEM-EDS composition results of the FeCoNiCrMn and FeCoNiCrMn-Al specimens (all in at%).

Elements	Fe	Co	Ni	Cr	Mn	Al
FeCoNiCrMn	19 ± 2	20 ± 1	22 ± 3	20 ± 1	19 ± 2	–
FeCoNiCrMn-Al	17 ± 2	19 ± 1	20 ± 3	19 ± 1	17 ± 2	8 ± 1

3. Results and discussionora

3.1. X-ray diffraction analysis

Fig. 2(a) presents the XRD scans of the as-cast and SMATed FeCoNiCrMn samples. For the SMATed sample, XRD was performed on the plane already subject to SMAT bombard. They can well be indexed by the FCC crystal structure, with a lattice parameter of 0.362 nm. For both the as-cast and SMATed samples, the (111) planar texture appears to be remained. But by careful measurements, the peak height ratio of (111) peak versus (200) peak seems to be lowered, from 8.0 for the as-cast sample down to 4.5 for the SMATed sample. This suggests that the FCC grain orientation distribution in the SMATed sample has become more random by the SMAT deformation.

In comparison, Fig. 2(b) shows the XRD results of the as-cast and SMATed FeCoNiCrMn-Al samples, which are of the duplex phase with the FCC (with a lattice parameter of 0.362 nm) and BCC structure (with a lattice parameter of 0.282 nm). The volume fraction of the FCC and BCC phases is ~80% and ~20%, respectively, based on the summation of the all FCC and BCC integral peak intensity. From the XRD (111) and (200) FCC peak ratio, the as-cast and SMAT samples appears to possess similar FCC texture. This FCC + BCC two-phase structure in the as-cast sample appears to be more difficult to be altered by SMAT, due to mutual restriction for grain orientation rotation. Finally, the peak widths increase apparently, suggesting strong grain size refinement and the presence of atomic-level lattice strain.

3.2. EDS and SEM analyses

All cast alloys were confirmed for their resulting compositions by SEM/EDS. Table 1 lists the results. Though there is scattering, the alloy compositions still fall into the range initially designed.

Fig. 3 presents the SEM micrographs taken form the cross sections of the SMATed FeCoNiCrMn and FeCoNiCrMn-Al specimens, at three different depths from the free surface that was subject to SMAT