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Paper Title: Effects of deformation-induced martensitic transformation on cryogenic fracture toughness for metastable Si8V2Fe45Cr10Mn5Co30 high-entropy alloy

Content :

1. Introduction

Great attentions have been made to improve mechanical properties of high-strength structural alloys used in cryogenic applications [1], [2], [3], [4], [5]. In this regard, face-centered-cubic(FCC)-based high- and medium-entropy alloys (HEAs and MEAs) such as equi-atomic CrMnFeCoNi or CrCoNi alloys have been developed as they possess excellent cryogenic properties due to a twinning-induced plasticity (TWIP) in both alloys or transformation-induced plasticity (TRIP) from FCC to hexagonal-close-packed (HCP) martensite in the CrCoNi alloy [6,7]. An enormous amount of TRIP from FCC to body-centered-cubic (BCC) was also found in a $V_{10}Fe_{50}Cr_{10}Co_{30}$ (at.%) alloy exhibiting an enhanced strain-hardening rate [8]. This FCC-BCC TRIP effect resulted in a very high tensile strength of 2 GPa at cryogenic temperature, although the yield strength and ductility were not sufficient in cryogenic applications.

Recently, a non-equi-atomic $Si_xV_{(9-x)}Cr_{10}Mn_5Fe_{46}Co_{30}$ FCC-based alloy system was developed by delicately controlling relative phase stabilities based on equilibrium phase diagrams and Gibbs free energies [9]. In an as-annealed state, a transformation from FCC to HCP appeared as the stability of FCC decreased with increasing Si content [9]. Under the tensile deformation, the further transformation occurred from FCC to BCC *via* intermediate HCP, producing a tensile strength beyond 1 GPa at room temperature [9]. Although the tensile properties of these alloys are outstanding, it is essentially necessary to investigate the fracture toughness representing the reliability in cryogenic environments. The cryogenic fracture toughness often deteriorates by a reduction of damage-tolerance capacity as a ductile-brittle transition (DBT) phenomenon might occur by a considerable amount of the transformed BCC martensite [10], [11], [12]. Nevertheless, a correlation of macroscopically measured fracture toughness with microscopic fracture mechanisms related with the transformed BCC martensite has hardly been verified yet.

In this study, the fracture toughness of the $Si_8V_2Fe_{45}Cr_{10}Mn_5Co_{30}$ alloy was measured at room and cryogenic temperatures, and the data were explained by deformation and fracture mechanisms in comparison with those of previously reported $V_{10}Fe_{45}Cr_{10}Co_{30}Ni_5$ [13], $V_{10}Fe_{45}Cr_{10}Co_{20}Ni_{15}$ [14], CrMnFeCoNi [6], and CrCoNi [7] alloys. Microfracture processes and fractographic observations were examined by revealing how the martensitic transformation triggered microcracks at the crack tip area under a relatively low applied stress field [15], [16], [17], [18], [19]. These results were explained by a morphology of transformed martensite varied with deformation mechanisms and stability of FCC phase.

Tensile properties

Fig. 3a shows engineering stress-strain curves at 25 and −196 °C of the $Si_8V_2Fe_{45}Cr_{10}Mn_5Co_{30}$ alloy, and the measured properties are summarized in Table 1 along with those of $V_{10}Fe_{45}Cr_{10}Co_{30}Ni_5$ [13], $V_{10}Fe_{45}Cr_{10}Co_{20}Ni_{15}$ [14], CrMnFeCoNi [6], and CrCoNi [7] alloys. At 25 °C, the yield and tensile strengths are 424 and 1099 MPa, respectively, while the elongation is 52%. At −196 °C, the yield and tensile strengths increase dramatically over those at 25 °C, while the elongation decreases slightly because of the active TRIP from FCC to HCP and BCC and resulting high strain-hardening effect [23]. The tensile strengths at both 25 and −196 °C are higher than those of the other alloys, as shown in Table 1. Fig. 3b shows strain-hardening-rate curves at 25 and −196 °C. A down-up-down curve shape is shown at both temperatures, and overall strain-hardening rate is higher at −196 °C than at 25 °C.

Fracture toughness

Fig. 3c shows crack-growth-resistance (J_R) curves composed of J-integral as a function of crack extension (Δa) at 25 and −196 °C. Critical value of J (J_C) is determined from an intersection point between J_R curve and blunting (dotted) line, while the plane strain condition, *i.e.*, B and b_0 >> 10 J_Q/σ_{flow} (B: specimen thickness, b_o: un-cracked ligament, σ_{flow}: (yield strength + tensile strength)/2)), satisfies at 25 and −196 °C. Critical plane-strain value of J (J_{Ic}) is 411 ± 20 and 76 ± 5 $kJ{\cdot}m^{-2}$ at 25 and −196 °C, respectively. Elastic modulus (E) and Poisson's ratio (ν), measured from a resonance-ultrasound spectroscopy paired with a laser-doppler interferometry in a vacuum at a constant temperature [25,37], are 194.9 GPa and 0.239, respectively, at 25 °C, and 207.4 GPa and 0.229, respectively, at −196 °C. Critical plane-strain value of stress intensity factor (K_{JIc}) converted from J-K relationship [24] is 292 ± 7 and 129 ± 4 $MPa{\cdot}m^{1/2}$ at 25 and −196 °C, respectively. The K_{JIc} at −196 °C is much lower than that at 25 °C despite of the higher yield and tensile strengths (Fig. 3a, Table 1). When the J_{Ic} and K_{JIc} of the present alloy are compared with those of the other alloys in Table 1, they are the highest at 25 °C, whereas they are the lowest at −196 °C.

Fig. 6a-e shows SEM fractographs of the fracture initiation area located in front of the fatigued-pre-crack of the CT specimens. The pre-crack is indicated by yellow arrows. At 25 °C, a ductile-dimpled fracture mode prevails in front of the pre-crack (Fig. 6a). In the magnified fractograph of the ductile-dimpled area of Fig. 6a, the fracture surface consists of a number of dimples, and some of them contain V-rich oxides as indicated by white arrows (Fig. 6b). This is because some large dimples form by initiating voids at the oxides [6,7,38]. At −196 °C, ductile-dimpled and quasi-cleavage areas appear in the near-crack-tip area (Fig. 6c). Fig. 6d,e shows magnified fractographs of ductile-dimpled and quasi-cleavage areas of Fig. 6c, respectively. In the ductile-dimpled area, dimples seem to be smaller and shallower than those found at 25 °C (Fig. 6d), and are partly mixed with quasi-cleavage areas. The quasi-cleavage area of Fig. 6e contains many parallel lamellar facets. These shallow dimples and lamellar cleavage facets are causative of the lower fracture resistance at −196 °C than at 25 °C.