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Thermal vacancy behavior analysis through thermal expansion, lattice parameter and elastic modulus measurements of B2-type FeAl

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Abstract

Thermal vacancy behavior in B2-type FeAl was analyzed through thermal expansion, lattice parameter, and elastic modulus measurements. High-temperature X-ray diffractometry (HT-XRD) was conducted to determine the lattice parameter at elevated temperatures, and the electromagnetic acoustic resonance method was applied to investigate the temperature dependence of the elastic moduli in B2-type FeAl. Using a series of in situ high-temperature techniques such as HT-XRD and dilatometry, the thermal vacancy concentration at elevated temperatures was estimated from the divergence between the changes in the sample length and the lattice parameter with temperature, giving a vacancy formation enthalpy of ~0.7 and 0.6 eV for Fe–40Al and Fe–43Al (at.%), respectively. The long-range order parameter was found to increase with temperature in a high-temperature range, indicating that the Fe-atom recovery process occurs in this temperature range. The in situ high-temperature measurements suggest that at elevated temperatures, thermal vacancies have no significant influence on the lattice parameter and elastic moduli of B2-type FeAl.

Keywords: Iron aluminides; Vacancies; X-ray diffraction; Lattice parameter; Elastic moduli

1. Introduction

As aluminum and iron are the two most commonly used metallic resources, iron aluminides have been widely studied as structural materials because of their interesting performance at high temperature. B2-type FeAl is one such representative intermetallic compound, and the thermal vacancy behavior and positive temperature dependence of the yield strength, i.e., the so-called strength anomaly, have been the two principal research topics for B2-type FeAl in the past few decades. It is well known that this intermetallic compound contains a high concentration of thermal vacancies at high temperature and that these vacancies are easily frozen into the material upon cooling [1,2]. These quenched-in vacancies lead to "excess vacancy hardening" at room temperature. It was first reported by Rieu and Goux [1] that rapid quenching from an elevated temperature significantly enhanced the hardness of B2-type FeAl due to supersaturated vacancies. Nagpal and Baker [3] reported that different cooling rates had a dramatic effect on the hardness of FeAl, while Chang et al. [4] demonstrated that over a large composition range (40–51 at.% Al) the changes observed in the hardness with increasing aluminum content and quenching temperature were an indication of the relative changes in the concentration of the supersaturated vacancies in FeAl. Moreover, supersaturated vacancies cause an increase in the critical resolved shear stress and a decrease in the ductility of single crystals at room temperature [5]. Through a study on the relationship between the vacancy concentration and the yield strength of FeAl with varying aluminum contents, Xiao and Baker [6] showed that vacancies control the room-temperature mechanical properties of FeAl. The excess vacancy concentration, however, can be reduced by intermediate temperature annealing, and Nagpal and Baker

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[3] demonstrated that the hardness of furnace-cooled FeAl reduced after annealing at 400 °C for 118 h. Yoshimi et al. [7] also showed that after being completely annealed at 425 °C for 120 h, the hardness of Fe–48Al (at.%) was even lower than that of Fe–39Al. Due to the removal of supersaturated vacancies, exothermic peaks were observed in differential scanning calorimetry (DSC) curves of quenched FeAl [8,9].

Several studies have investigated the behavior of thermal and quenched-in vacancies to understand their effects on the mechanical properties of B2-type FeAl. Here, it is worth mentioning the "vacancy hardening" model, which was proposed by George and Baker [10] to explain the strength anomaly of B2-type FeAl at elevated temperatures. Because the vacancy formation enthalpy is much lower than the migration enthalpy [11], thermal vacancies in FeAl form easily but find it difficult to migrate. In their theory, newly generated thermal vacancies in the strength anomaly temperature range can hardly move and so impede the dislocation motion by the "solid solution" hardening mechanism. Since the positive temperature dependence of the yield strength in B2-type FeAl was found by Chang [12] in 1990, a number of mechanisms have been proposed to explain the phenomenon. In addition to the vacancy hardening model, both "glide decomposition" [13] and "local climb locking" [14] are also candidate models. However, the discussion about the strength anomaly in B2-type FeAl is far from finished.

In this study, thermal expansion, lattice parameter and elastic modulus measurements were conducted to study the thermal vacancy behavior in B2-type FeAl. Several samples subjected to different thermal histories were examined at room and elevated temperatures, and the thermal vacancy concentration and its formation properties were estimated. In light of the obtained results, we discuss excess vacancy hardening and the role of thermal vacancies on the temperature dependence of the lattice parameter and elastic moduli.

2. Experimental procedures

Ingots with compositions of Fe–40Al and Fe–43Al (at.%) were prepared by conventional argon arc-melting using pure aluminum (99.99 wt.%) and pure iron (99.99 wt.%). Each ingot was melted five times, flipping the ingot upside down after each melt to ensure homogeneity. The as-cast ingots were subjected to a homogenization heat treatment at 1100 °C for 24 h under vacuum conditions of better than 10^{-3} Pa and then slowly cooled to 400 °C, before being aged at the same temperature for 100 h to eliminate supersaturated vacancies. The ingots were then furnace-cooled to room temperature. All the experimental specimens were prepared from these fully annealed ingots.

Powder samples for X-ray diffraction (XRD) measurements were prepared by crushing parts of these ingots in a tungsten mortar. The size of the powder particles was less than 46 μm in diameter. The powder was sealed in a quartz tube under vacuum conditions of better than 10^{-2} Pa and heated at 800 °C for 1 h to eliminate the residual strain followed by the excess-vacancy-elimination heat treatment at 400 °C for 100 h. All the powder samples described below were subject to this thermal history. The lattice parameters of the FeAl powder samples were measured at both room and elevated temperatures using Co Kα X-rays and a D8 ADVANCE (Bruker AXS) diffractometer equipped with a HTK1200N high-temperature furnace (Anton Paar). For the room-temperature measurements, the powder was first sealed in the quartz tube, filled with 4×10^4 Pa Ar gas and quenched at different temperatures. For the high-temperature measurements, the powder was kept under vacuum conditions on the order of 10^{-3} Pa at the corresponding testing temperature for 30 min before each test to avoid temperature inhomogeneities. Oxidation was not detected in the samples since no oxide peak appeared in any of the measurements up to 1000 °C. The lattice parameter values were estimated from the whole powder pattern decomposition (WPPD) method, and the integrated intensity of the diffraction peaks was calculated by the single profile fitting (SPF) method using the TOPAS4 software (Bruker AXS). The long-range order parameter was estimated from the integrated intensity ratio of the superlattice and fundamental diffractions in the XRD profiles.

Several bulk specimens were also cut from the fully annealed ingots by electrodischarge machining (EDM). These specimens were used for hardness, thermal expansion and elastic modulus measurements. The thermal expansion of the 5 mm × 5 mm × 20 mm samples was measured by thermomechanical analysis (TMA) using a Q400 analyzer (TA Instruments Japan Inc.). Isothermal measurements were conducted by keeping the temperature of the specimens fixed for 30 min at every 100 °C interval between 100 °C and 1000 °C to provide the same heating path as that in the high-temperature XRD measurements. The dimension changes of the specimens were taken as the average of the values in the last 10 min at each temperature step. The thermal vacancy concentration was estimated from the divergence between the lattice parameter change and the thermal expansion of the samples. Moreover, the activation energy of vacancy formation was derived using the Arrhenius relationship. The temperature dependence of the elastic moduli was obtained using the electromagnetic acoustic resonance (EMAR) method using a CCII-HT instrument (Nihon Techno-Plus Co. Ltd.). As shown schematically in Fig. 1, a 5 mm × 5 mm × 5 mm specimen surrounded by a coil was placed between two hard magnets. Two different vibration modes were excited in the specimen by the Lorentz force when an electric pulse was sent through the coil, and the ultrasonic waves returned to the coil were transformed into digital signals. The moduli measurements were performed at room temperature to up to 800 °C over a frequency range of 350–1200 kHz in a magnetic field of 0.5 T. The specimens were