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Influence of heat treatment on the microstructure and hydrogen storage properties of $Ti_{10}V_{77}Cr_6Fe_6Zr$ alloy

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ABSTRACT

The as-cast $Ti_{10}V_{77}Cr_6Fe_6Zr$ alloy was heat-treated at 1373 K for 8 h or 1523 K for 5 min and then quenched in water. The influence of heat treatment on the microstructure and hydrogen storage properties of $Ti_{10}V_{77}Cr_6Fe_6Zr$ alloy was investigated systematically. The results show that all of the as-cast and heat-treated alloys consist of BCC main phase and C14 Laves secondary phase. After heat treatment, the phase abundance of BCC enhances and the plateau region of P–C–T curve is flattened, but the hydrogen absorption capacity is decreased. However, the alloy heat-treated at 1523 K for 5 min achieves a enhanced hydrogen desorption capacity of 1.82 wt.% at 333 K against 0.1 MPa, which is higher than 1.44 wt.% hydrogen desorption capacity of the as-cast alloy.

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1. Introduction

Vanadium or vanadium-based solid solutions, as the third generation of hydrogen storage alloys, have been widely studied and several series of V-based multi-component alloys with good hydrogen storage properties have been developed. Among which Ti–Cr–V alloys attracted much more attention for its high capacity of absorbing hydrogen and excellent kinetics for hydrogen absorption and desorption at moderate conditions [1–4]. However, these alloys exists some shortcomings, such as difficulty in activation, poor P–C–T plateau characteristics and low hydrogen desorption capacity for the much lower dehydrogenation pressure plateau region of mono-hydride (e.g. plateau pressure of about 1 Pa at room temperature for the hydrogen desorption of VH) [3–8].

It is well known that the partial substitution of V with other transition elements, such as Fe, Zr and Mn, is a very effective way to improve the overall hydrogen storage properties and lowering their cost of the Ti–Cr–V alloys [9,10,6,11–23]. On the other hand, it was reported that the heat treatment could also impact the microstructure and hydrogen storage properties of the Ti–Cr–V alloys [3,24,25]. Akiba and Iba [3] reported that the hydrogen storage properties of Ti–Cr–V alloys in as-cast state were sensitive to heating, therefore it was reasonable to believe that the heat treatment might affect the hydrogen storage properties of these alloys. Okada et al. [24] investigated the effect of heat treatment on the hydrogen storage properties of the Ti–Cr–V alloys, and reported that moderate heat treatment could enhance the hydrogen storage capacity and flatten the hydrogen desorption pressure plateau, e.g. the $Ti_{25}Cr_{40}V_{35}$ alloy annealed at 1573 K for 1 min and then quenched in water achieved a hydrogen desorption capacity of 2.4 mass% at 313 K against 0.01 MPa. Cho et al. [25] also reported that $Ti_{32}Cr_{43}V_{25}$ alloy annealed at 1653 K for 1 min achieved a hydrogen desorption capacity of 2.3 mass% at 303 K against 0.001 MPa. However, the influence of the heat treatments with the relatively lower temperature and longer time (such as 1523 K for 5 min or 1373 K for 8 h) on the microstructure and hydrogen storage properties was not reported. In this paper, the corresponding heat treatment conditions (annealed at 1523 K for 5 min and 1373 K for 8 h) were employed for $Ti_{10}V_{77}Cr_6Fe_6Zr$ alloy which was explored and optimized in our previous work [22,23], and the influence of heat treatment on the microstructure and hydrogen storage properties of this alloy was investigated in detail.

2. Experimental

The as-cast $Ti_{10}V_{77}Cr_6Fe_6Zr$ alloy was prepared by levitation induction melting under argon atmosphere, and the ingot was turned over and remelted four times to ensure its homogeneity. Then two methods of heat treatment were adopted: (1) annealing at 1373 K for 8 h and then quenching in water; (2) annealing at 1523 K for 5 min and then quenching in water.

In order to investigate the phase structure, the morphology and the phase composition of the alloy, X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM), and energy dispersive X-ray spectroscopy (EDS) were performed, respectively.

For activation procedure, the sample of 4 g was placed in the reactor of Sieverts type apparatus and evacuated for 15 min under room temperature firstly, and then

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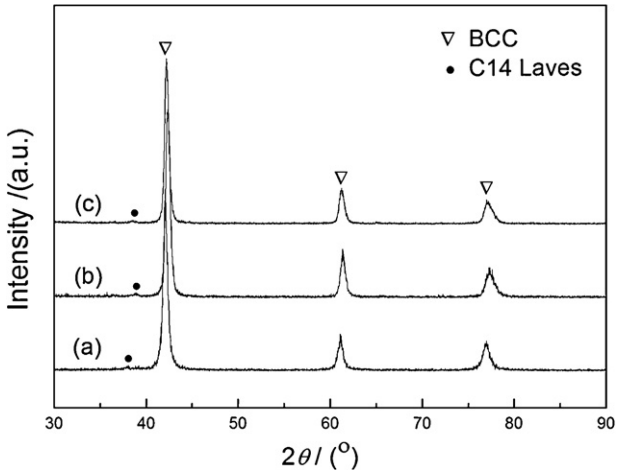

Fig. 1. XRD patterns of the as-cast and heat-treated $Ti_{10}V_{77}Cr_6Fe_6Zr$ alloys: (a) as-cast; (b) heat-treated at 1373 K for 8 h and then quenched in cold water; and (c) heat treated at 1523 K for 5 min and then quenched in cold water.

hydrogen was introduced gradually into the reactor up to a pressure of 4 MPa for the absorption process. Also the hydriding testing was performed under room temperature with a starting pressure of 4 MPa. The dehydriding kinetics curves were obtained against 0.1 MPa at 333 K. After dehydriding, the reactor was evacuated for 30 min at 673 K to extract the residual hydrogen for the next hydriding process. After three hydriding-dehydriding cycling, the dehydrogenation P–C–T measurement were carried out at 333 K. The change in pressure with time was recorded. In this study, the effective hydrogen desorption capacity is defined as the amount of hydrogen desorbed when the hydrogen pressure is decreased from 3 MPa to 0.1 MPa. Differential scanning calorimetry (Netzsch STA 449F3) measurements of the samples were performed under the pressure of 0.1 MPa with a constant heating rate of 10 K/min. The powder size used for the XRD and DSC measurements is under 30 μm.

3. Results and discussion

Fig. 1 shows the XRD patterns of the studied alloys. It can be seen from Fig. 1 that the as-cast alloy consists of BCC main phase and C14 Laves secondary phase. After heat treatment, the intensity of diffraction peak of the BCC phase enhances, while that of C14 Laves phase becomes weak, which implies that these heat treatments promote the growing of the BCC phase. The diffraction peak shifts to higher angle and lower angle for samples b and c, respectively. The lattice parameters of the BCC main phase for the studied alloys were determined by the Retvield refinement

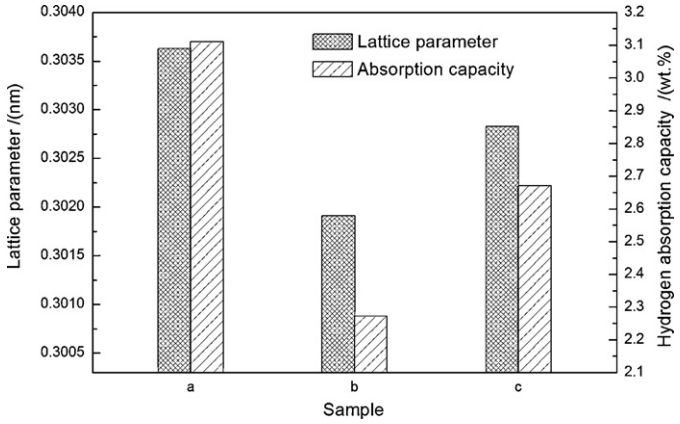

Fig. 2. The relationship between heat treatment conditions and lattice parameters and hydrogen absorption capacities: (a) as-cast; (b) heat-treated at 1373 K for 8 h; and (c) heat-treated at 1523 K for 5 min.

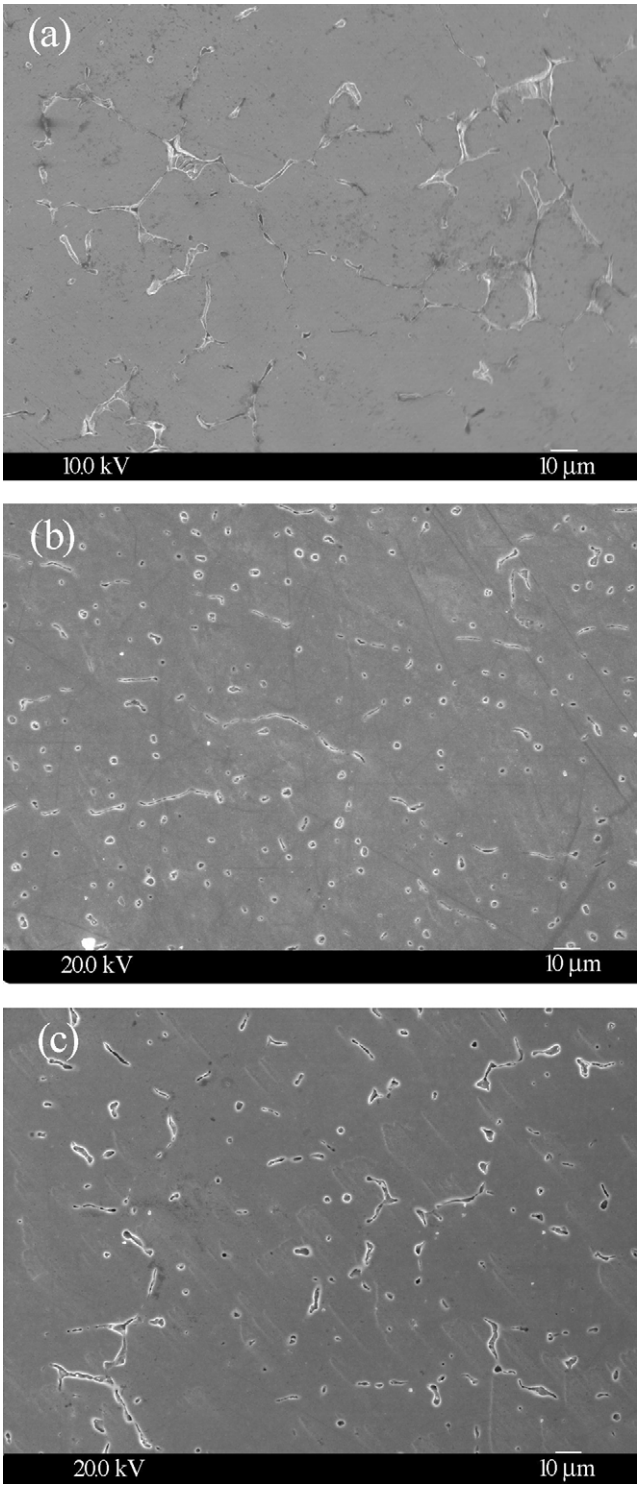

Fig. 3. SEM micrographs of the as-cast and heat-treated $Ti_{10}V_{77}Cr_6Fe_6Zr$ alloys: (a) as-cast; (b) heat-treated at 1373 K for 8 h; and (c) heat-treated at 1523 K for 5 min.

analyses and calculated as 0.30363 nm, 0.30191 nm and 0.30283 nm, which are shown in Fig. 2. The reason why the lattice parameter decreased by the heat treatment might be attributed to the change in the element contents of the BCC main phase after different heat treatments.

Fig. 3 shows the SEM micrographs of the as-cast and heat-treated $Ti_{10}V_{77}Cr_6Fe_6Zr$ alloys. It can be observed that all the alloys consist of two phases, which is in good agreement with the results

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