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Research Letter

Growth of Functional FeTi Clusters Covered with Carbon Layer

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FeTi clusters with a diameter of less than 10 nm and covered with a graphitic layer have been preferentially produced in an H_2 gas atmosphere at pressures of 10 and 26.6 kPa by the simultaneous evaporation of Fe and Ti wires from a concave carbon boat. To compare this result with cluster formation in an inert gas atmosphere, the result for an Ar gas pressure of 10 kPa is also discussed. The formation of disordered FeNi clusters predominately took place in an H_2 gas atmosphere.

1. INTRODUCTION

Hydrogenis an ideal fuel for all types of fuel cell. It can be stored as a compressed gas, a liquid, and solid as a combined with other elements. For hydrogen storage, the ideal type of hydride is a metal or alloy hydride that reversibly absorbs and desorbs hydrogen. A typical example is FeTi alloy: $FeTi + H_2 \rightarrow FeTiH_2$ [1]. The range of pressures of interest for a hydrogen storage system in fuel cell applications is about (1 to 5) $\times 10^5$ Pa. Also, it is necessary for hydrogen desorption to occur below 100°C. FeTi and VNb alloys are potentially suitable alloys. Alloys are generally preferentially produced by the mechanical alloying method. The oxide layer on a metallic powder surface is a practical problem during the alloy formation. Carbon nanotubes may also be good hydrogen absorbers [2]. Since nanotubes are based on the graphite structure, nanographitic carbon particles may also act as a hydrogen absorber. Most metallic particles of the order of 50 nm can be produced by the gas evaporation method in an inert gas [3]. Metallic clusters covered with an insulator layer of SiO_2 or an electrically conductive carbon layer can be produced by the advanced gas evaporation method [4, 5].

In this paper, a new production method for the formation of FeTi clusters of less than 10 nm diameter covered with a carbon layer has been reported. In the present study, hydrogen gas has been used to replace the inert gas.

2. EXPERIMENTAL

To produce the FeTi alloy, Fe and Ti wires of 5 mmϕ were inserted into a carbon rod with a concave shape as shown in Figure 1. The length of the concave region was 10 mm. Fe and Ti wires with a composition ratio of 50:50 atomic percent were inserted in the concave region. Two carbon rods were pushed together by springs inserted the stainless-steel electrodes as schematically shown in Figure 1(a). The work chamber used was a glass cylinder 17 cm in diameter and 30 cm in height, covered with stainless steel plate, and connected to a high vacuum exhaust through a valve at its bottom. The preset method is very similar to the arc evaporation method of carbon [6]. To compare the produced materials with those obtained by inert gas evaporation, the Fe and Ti wires were evaporated in Ar gas at 10 kPa and hydrogen gas at 10 and 26.6 kPa. The collected samples were examined using a Hitachi H-9000NAR electron microscope.

3. RESULTS AND DISCUSSIONS

Figure 2 shows a typical electron microscopic (EM) image and the corresponding electron diffraction (ED) pattern for the samples produced in Ar gas at a pressure of 10 kPa. The ED pattern shows the formation of a mixture of Fe, Ti, FeTi, and TiC phases. The large particles in the EM image are Fe

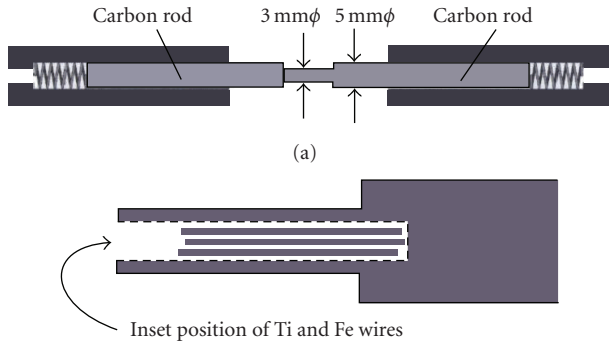

Figure 1: Schematic representation of present production method. (a) Two carbon rods were pushed together by springs. (b) Concave region of 3 mmϕ as in the diagram. Ti and Fe wires were set in the concave region.

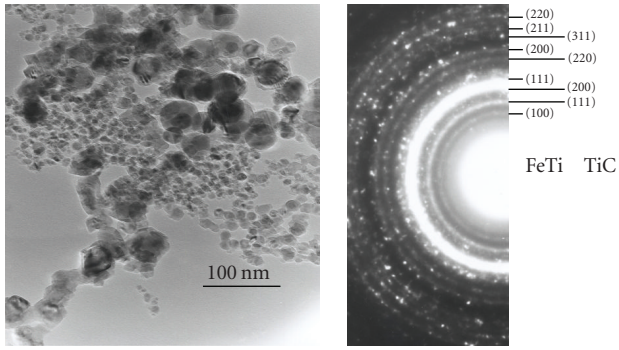

Figure 2: EM image and corresponding ED pattern of the particles produced in Ar gas pressure of 10 kPa. The spotty diffraction spots were Fe and Ti particles. Rings were indexed as FeTi and TiC nanoparticles, which are indicated in (b). An FeTi ordered phase appeared.

and Ti particles [3]. The Ti particle surface was covered with a TiC layer [7]. Each particle was covered with a carbon layer. The FeTi particles were in an ordered phase.

When the same experiment was performed in hydrogen-gas, the collected particles were indexed as the disordered FeTi phase as shown in Figure 3. The dark field image clearly shows that the FeTi cluster formation is less than 10 nm in size. Figure 4 shows a high-resolution transmission electron microscopic (HRTEM) image of an FeTi nanoscale particle covered with a graphitic carbon layer. The preferential formation of the FeTi disordered phase in hydrogen gas may be due to the fact that Fe, Ti, and FeTi are typical hydrogen absorbing materials, and that Fe and Ti particles produced in Ar gas, as shown in Figure 2, were destroyed by the absorption of hydrogen resulting in the formation of more stable FeTi. Using the smoke particle formation process, growth by coalescence among the particles predominately occurs [8]. Thus, stable FeTi nanocrystallites can be produced. When the hydrogen pressure was 10 kPa, the FeTi crystallites were slightly larger than those formed at 26.6 kPa, as recognized by

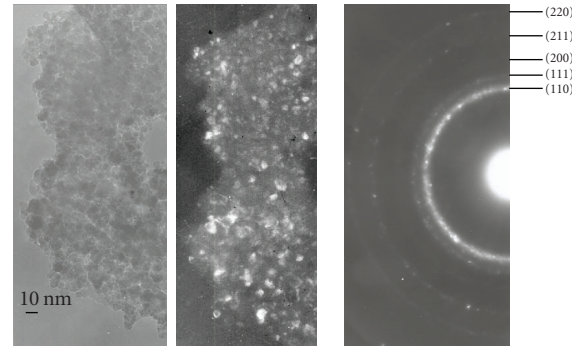

Figure 3: EM images ((a) bright and dark field) and corresponding ED pattern of the specimen produced in H_2 gas at 26.6 kPa. Nanocrystallites of diameter less than 10 nm were clearly seen in dark field image. (b) The ED pattern can be indexed by disordered FeTi. The appearance of (111) suggests that the FeTi nanoparticles may include hydrogen atoms.

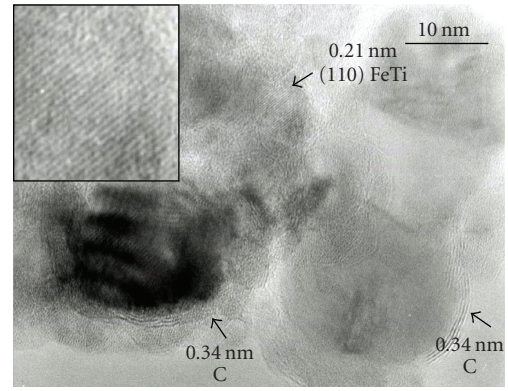

Figure 4: HRTEM image of FeTi particles produced in H_2 gas atmosphere. Particles were covered by a graphitic carbon layer as indicated by arrows. The left top enlarged image of (110) FeTi shows the Guinier-Preston zone-like contrast [8]. This suggests that hydrogen atoms are dissolved in the FeTi crystal.

the sharpness of the diffraction pattern. Growth of the FeTi particles of the order of 10 nm was preferentially observed. This result also suggests that the hydrogen gas restricted the formation and the growth of alloy particles. The alloy clusters produced in the H_2 gas atmosphere were covered with a carbon layer, thus preventing oxidation of the metallic alloy clusters. Fundamental experiments on hydrogen absorption are now being carried out by another laboratory group. The results will be presented elsewhere. The absorbent alloy formed by the present method is expected to be used as another hydrogen permanent absorbent material. A systematic study on the formation of the alloy is now being carried out by our laboratory group, and the results will be published elsewhere.

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