Ti-Cr Nanoparticles Prepared by Electrical Wire Explosion

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We previously proposed a new method to synthesize nano-sized powders of various alloys and intermetallic compounds. The method consisted of electrical wire explosion of electrodeposited metal wires. In this study, the method was applied for Ti-Cr alloy. The overall composition of the Cr-coated Ti wire was about Ti-25 at% Cr. The explosion products consisted of equilibrium phases of α-Ti and TiCr₂ phases along with meta-stable Ti-rich β phase. The composition of β phase estimated from its lattice parameter was Ti-13 at% Cr. The β phase decomposed completely to α-Ti and TiCr₂ phases during isothermal aging at 600°C for one day. [doi:10.2320/matertrans.M2009190]

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1. Introduction

The electrical wire explosion (EWE) has been considered as one of the practical means to the production of nano-sized particles of pure metals. However, the method was not very successful in the production of alloys or intermetallics due to the unavailability of brittle wires of corresponding composition. As a result, synthesis of alloy nano particles by EWE has been restricted to commercially available alloys as AlSi, Cu-Zn, Ni-Ti, Cu-Ni. We tried to circumvent the restriction by employing coated wires instead. By the method, we could synthesize single phase alloy nano particles of Cu-Ni and Cu-Ni-P alloys which exhibit complete mutual solubility. In this study, we apply the method to Ti-Cr alloys which have a eutectoid reaction.

Earlier studies on Ti-Cr alloys were focussed on the omega phase in connection to its effect on mechanical properties. It has been well established that the meta-stable bcc β phase can easily be retained by a moderate rate of quenching from β-phase region owing to the sluggish eutectoid transformation. The interest for Ti-Cr alloy has been renewed recently for its excellent hydrogen sorption and desorption properties. It has been shown that alloys of bcc structure can adsorb a large amount of hydrogen. It would be interesting to see whether nano powders having bcc β phase could be produced in Ti-Cr system. In this study, we report the synthesis of Ti-Cr alloy nano powders using the previously suggested method.

2. Experimental Procedure

The Ti-Cr wire for EWE was prepared by the electrodeposition of Cr on 0.289 mm Ti wire. The detailed procedure for the wire coating set-up is described in previous papers. The final diameter of the Cr-coated Ti wire was 0.320 mm which gives the average composition of about 25 at% Cr. The Cr-plating solution consisted of 2.47 mol/L of chromic trioxide and 0.03 mole/g of sulfuric acid. The temperature of deposition was maintained at 40°C. The cell voltage was 5 volt and the current density was about 10 mA/cm². Pure lead plate was used for the anode. The plating conditions were the same throughout the experiment and the thickness of the Cr coating was adjusted by the pulling speed through the plating bath. The Cr-coated Ti wire was then loaded to the chamber for explosion experiments.

The capacitance of the exploding circuit was 3.5 µF. The applied voltage across the 2 cm-long wire was 11.4 kV. The explosion was conducted under argon pressure of 2 atm. After explosion, it was stabilized overnight and then classified by 120 mesh sieve to remove the misfired portions of the Cr-plated Ti wires and residues. The particles were examined by Scanning Electron Microscopy (SEM), Field Emission Transmission Electron Microscope (FE-TEM), and X-ray Diffraction (XRD) with Cu Kα radiation. The composition was measured by EDS attached in SEM and FE-TEM.

3. Results and Discussion

The exploded particles have a spherical shape. Figure 1 shows the SEM micrographs of powders taken at different magnifications. The explosion products contained some large spherical powders ranging 2-20 µm (Fig. 1(a)). These particles are obviously formed from liquid and typical for the wire-explosion method. Few EDS measurements on individual particles showed that those are richer in Ti ranging 2-30 at% Cr. Higher magnification SEM photograph (Fig. 1(b)) shows clusters of spherical nano-sized particles of few tenths nanometers. Figure 2 shows a FE-TEM micrograph of the particles. It appears that the particles consist of two size groups, fine and coarse particles. Since the fine particles were smaller than 10 nm, it was difficult to measure the size on the photographs by conventional means. However, the average particle size of the coarser particles measured from several photographs was about 40 nm. Some of the coarser particles were often chemically inhomogeneous when examined by EDS. Currently, a more deliberate work on this aspect is underway in an effort to elucidate the formation mechanism of individual particles.

It is difficult to produce Ti or Ti-alloy nano particles of low oxygen content due to their high affinity for oxygen. The oxygen and nitrogen content of the Ti-Cr nano particles were 2.2 and 0.07 mass%, respectively. Figure 3(a)(b) shows the X-ray diffraction patterns of the explosion products. Here, (a) is the pattern of explosion products and (b) is that after
aging at 600°C for 1 day in vacuum. Before aging, the particles are laterally scattered on the bottom of a large alumina boat to minimize the possible mass transport between individual particles. Pure Ti wire pieces were also charged on the boat to reduce the oxidation of particles. The equilibrium phases corresponding to the average composition of the coated-wire, i.e. 25 at% Cr, are α-Ti and TiCr₂ phases.

The explosion products were α-Ti, bcc β(Ti,Cr) and TiCr₂ phases (Fig. 3(a)). Here, the TiCr₂ phases appear to be the mixture of three polymorphous types. The meta-stable β phase is retained as was frequently observed in case of bulk alloys. The wide diffraction peaks and spikes along the peaks appears to reflect their small particle sizes and the inhomogeneity in their composition. The β phase was completely decomposed to equilibrium phases of α-Ti and α-TiCr₂ phase during aging at 600°C for one day (Fig. 3(b)).

The lattice parameter of the bcc β phase calculated from the peak shown in Fig. 3(a) was 0.3254 nm. The composition of the β phase could be estimated from the results of previous studies. Figure 4 is the replot of previous reports and the lattice parameter can be expressed as

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\text{Lattice parameter (nm)} = 0.3310875 + 0.04354 \times \text{Cr (at.%)}
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Accordingly, the lattice parameter of β phase in this study corresponds to the Cr content of about 13 at%. This is about half of the average Cr composition of the starting wire. The lower Cr content of β phase may be due to the preferential formation of TiCr₂ phase on the initial stage of the explosion. In this study, the nucleation of the stable phases, e.g., TiCr₂, could not be suppressed completely as was the case of bulk samples where sluggish eutectoid reaction is involved. In
addition, it is known that oxygen stabilizes the α-Ti phase.\textsuperscript{26) If so then, the high oxygen content in Ti-Cr nanoparticles may also act to lower the stability of the β phase thus reducing the amount of β phase.

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REFERENCES