Internal Structure and Phase Transformation of Ti-Mo Alloy Fine Particles

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SYNOPSIS

Fine particles of Ti-Mo alloy have been prepared by means of arc method, and investigated on internal structure and phase transformation using HR-TEM and EDS. Martensite phase was observed in a particle containing comparatively low concentration of Mo, and ω phase was also found to exist in a nearly 14%Mo particle. The structure of the ω phase in the fine particle is expanded and remarkably unstable in comparison with the bulk sample, so that it has disappeared in a few seconds during TEM observation. Moreover, the β structure of Ti-Mo particles has changed to the unusual fcc phase with irradiation of a strong electron beam.

1. INTRODUCTION

There has been considerable interest in the phase transformation of ω phase and martensite in metastable β-Ti alloys[1-3]. Especially the ω phase is interesting in its marked embrittlment effect[4,5]. Both the ω phase and the martensite have been considered to be related to a strain field[3,6]. Many studies of the phase transformation in metastable β-Ti alloys have been concerned with the bulk samples. It has not been ascertained whether these phases could exsist in fine particles of metastable β-Ti alloys in which the effect of strain field is little in prospect. Reports on the fine particles of alloys are few[7,8] be-

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cause of difficulties in fabrication of particles containing a desired composition. For instance, in the case of producing fine particles of Ti-Mo alloy by means of gas evaporation method, the produced particles will not contain any Mo, because the vapor pressures of Ti and Mo are different to excess. We adopted arc method which was not so influenced by vapor pressure of each element in alloys. The present paper will describe internal structure and phase transformation in particles of Ti-Mo alloy investigated by a high resolution transmission electron microscopy(HR-TEM) and an energy dispersive X-ray spectrometry (EDS).

2. EXPERIMENTAL

The experimental apparatus to generate the fine particles is illustrated schematically in Figure 1. The specimen rods of Ti-20mass%Mo alloy were attached to both electrodes in a stainless steel vacuum chamber. The anode was movable to keep a constant clearance of about 0.5mm between the electrodes. The micro-grid to capture the particles was placed at 50mm above the electrodes. Fine particles of Ti-Mo alloy were evaporated by arc discharge between the electrodes for about 0.1s under 5.3×10^4 Pa in pressure of Ar. Because the arc generation for a

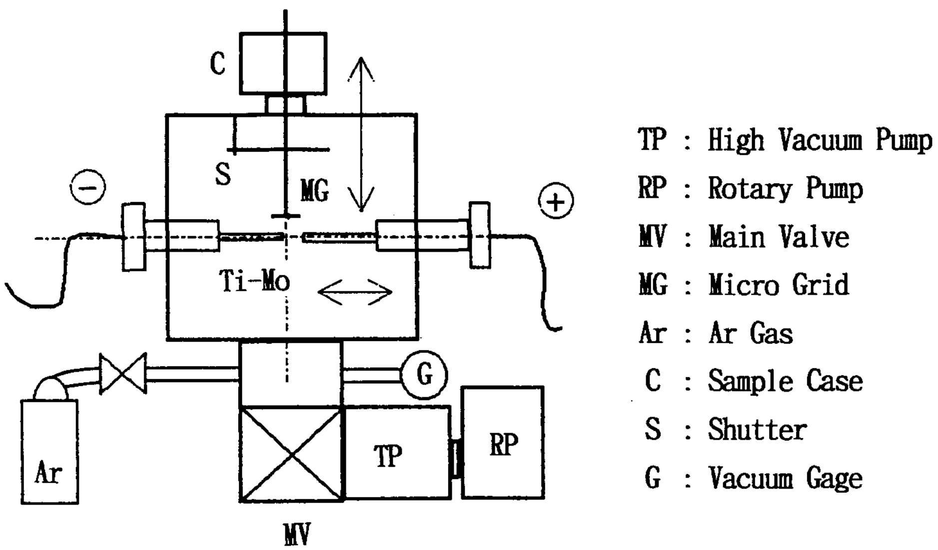

Figure 1. Illustration of apparatus to prepare the Ti-Mo alloy fine particles.

long time ($\sim$2s) makes the tip of specimen melt down, the particles formed therefrom will not contain any element of Mo as the evaporation method. The collection of fine particles was accordingly carried out by 20 times repetition of the short time discharge. HR-TEM images were taken using Topcon EM-002B operated at 200kV. The measurement of composition of individual particles was performed with Kevex EDS system and a thick Pt condenser aperture (360μm) to suppress generation of high energy Bremsstrahlung.

3. RESULTS AND DISCUSSION

3.1. Morphology of Ti-Mo Particles

Figure 2 shows the TEM images and the X-ray spectra of EDS obtained from the typical particles produced by the arc method. The features of each particle are generally expressed as follows :(a) the Ti particles are spherical, (b) the Ti-Mo alloy particles are nearly ellipsoidal and (c) the Mo rich particle is a polygonal huge ball. The particles in a group formed a chain, except for huge Mo balls.

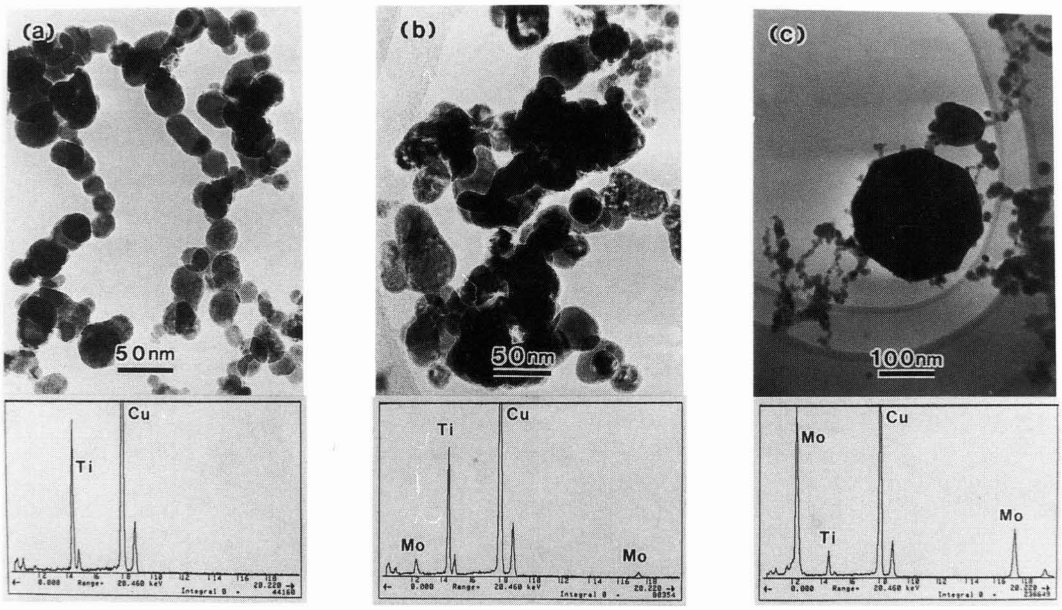

Figure 2. TEM images and X-ray stectra obtained from the typical particles, (a): Ti particles, (b): Ti-Mo particles and (c): an Mo rich particle.

In the EDS spectra, the peak of CuKα is generated from the Cu mesh of micro-grid, not related to the particles. Average composition of Ti-Mo particles generated by arc method is 13～15%Mo. The tendency of somewhat poor Mo as compared with the original specimen of electrodes is due to the difference of sputtering ratio between Ti and Mo.

Figure 3 shows the results of EDS analyses for each particle in a group. The particles organized into a group have nearly the same characteristics in size, shape and composition. It seems that the atoms in particles belonging to a group are sputtered from the same part on the original specimen of the electrode. The generation process of particles is schematically shown in Figure 4.

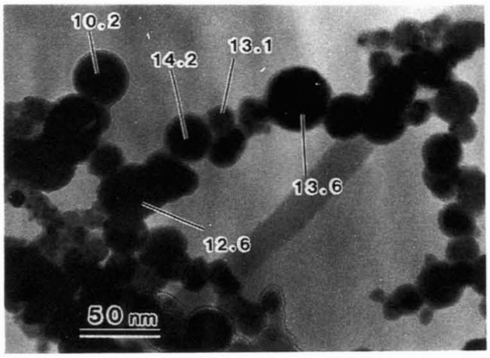

Figure 3. EDS analyses on each Ti-Mo particle in a group.

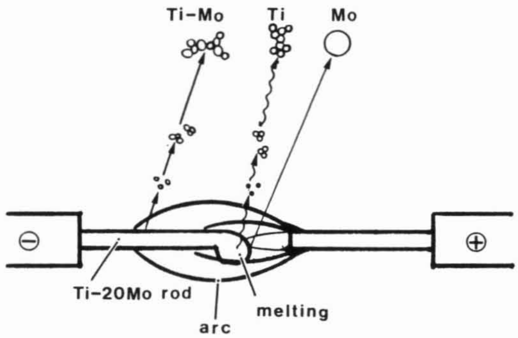

Figure 4. Schematic generation process of particles by arc method.

3.2. Internal Structure of Ti-Mo Alloy Particle

It is well known that the metastable β-Ti alloy quenched from the β field consists of β(bcc)+ω(trig.) phases, and becomes the stable phases such as β+α(hcp) by aging(1-3).

Figure 5 shows the selected area diffraction pattern (SADP) obtained from particles of about 14%Mo. The SADP rings can be mostly indexed as the β structure, although the rings of α phase are slightly recognized. It can be regarded as an effect of quenching through the formation process of particles.

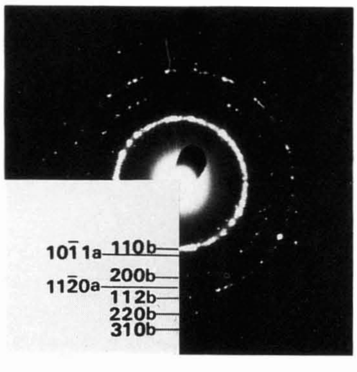

Figure 5. SADP from the particles of about 14%Mo.

Figures 6(a) and 6(b) show the HR-TEM images of particles of 3.3%Mo and 13.8%Mo, respectively. The particle in 6(a) has martensitic stacking fault fringes. Most of the martensitic particles observed have a low concentration of Mo ($\sim$5%Mo). On the other hand, the particles containing nearly 14%Mo occasionally has the ω like lattice fringes (d=0.40nm) as shown in 6(b). However, such a fringe may be regarded as Moiré fringes due to overlap of particles. The HR-TEM photograph should be taken from a single particle in order to ascertain existence of the ω phase in a particle. It was very difficult to take the photograph on TEM film, because the single particle was so easily drifted or shaken by irradiation of electron beam that the ω like lattice fringe in a single particle disappeared in a few seconds during TEM observation.

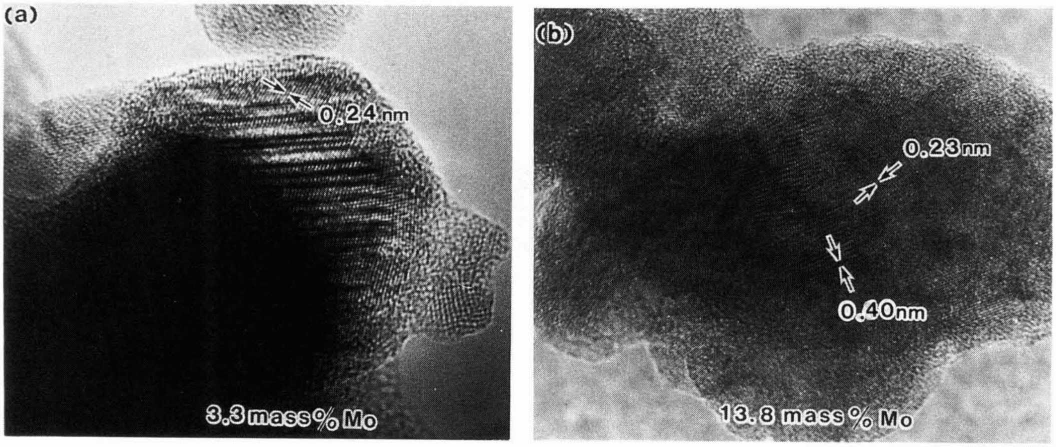

Figure 6. HR-TEM images of particles of (a):3.3%Mo and (b):13.8%Mo.

Therefore, HR-TEM image was recorded onto video tape using image intensifier. Figure 7(a) shows the processed video image of a single particle of Ti-Mo alloy indicating the ω like lattice fringe, and 7(b) shows the Fourier transformed pattern of the video image. The pattern of 7(b) is well explained by the assumption that one of the ω variants precipitates on the $\{113\}_\beta$ plane in the particle. However the ω phase in the particle is slightly different from the one in the bulk sample in regard to the lattice parameter. Namely the largest plane spacing of the ω which is 0.40nm in the bulk sample increases to 0.42nm in the particle. It is considerd that the constrained ω structure in the bulk is expanded and relaxed in the fine particle. From these results, it is evident that the metastable phases like ω and martensite can exist even in a fine particle of Ti-Mo alloy, however, the structure of the ω in a fine particle is remarkably unstable.

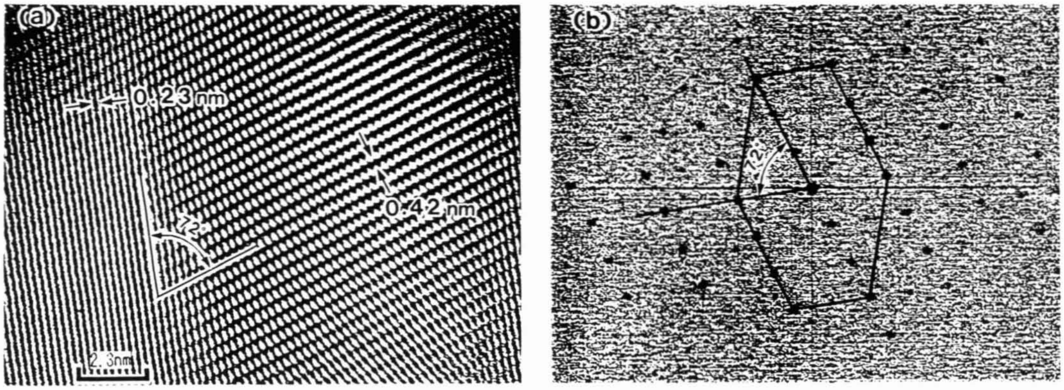

Figure 7. (a):Processed image of the ω like particle, and (b): Fourier transformed image.

The β structure of Ti-Mo particles is also unstable, and easily changes by irradiation of a strong electron beam. Figure 8(a) shows the SADP of Ti-Mo particles before the irradiation, and 8(b) and 8(c) show the SADP and TEM image of the identical particles after irradiation of a strong electron beam for 60 s by taking off the condenser aperture. It is apparently observed that the structure of particles changes from single β to fcc by the irradiation, and many white voids are yielded in the particles. The lattice parameter of the fcc phase has been determined by SADP as approximately 0.41nm. The fcc phases in Ti alloys which have been reported up to now are almost related to hydrides of Ti formed during electropolishing for preparation of TEM foils. In this case,the fcc phase in the particles is not related to the hydrides, but to the irradiation of a strong electron beam. It may be that the strong electron beam causes mixing of atoms, remelting and structure relaxation in the particles.

A study of the relation between the formation of fcc phase and the occurrence of many voids in the particles is currently in progress.

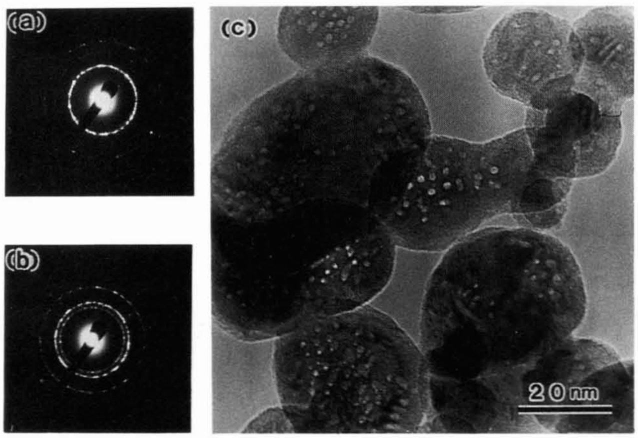

Figure 8. (a):SADP of Ti-Mo particles before the irradiation, (b) and (c): SADP and TEM image of the identical particles after the irradiation.

4. CONCLUSIONS

Fine particles of Ti-Mo alloy were produced by means of arc method, and were investigated on internal structure and phase transformation by HR-TEM and EDS. The particles containing about 14%Mo have ellipsoidal shapes and β structure. A group of particles in a chain consists of particles having the same characteristics in size,shape and composition Martensites were observed in the particles that have comparatively low concentration of $\sim$5%Mo, and ω phase was also found in the particle of nearly 14%Mo. It was noted that the irradiation of a strong electron beam changed the structure of particles from β to fcc, and generated many voids in the particles.

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