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Phase Evolution and Formation Process of Compound during Ball Milling of Ti-Si Powder Mixtures

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Introduction

It is believed that amorphous and crystalline compound powder are formed by ultrafine mixing of constituents and solid-state interdiffusion in mechanical alloying[1-5]. Recently, Atzmon[6] suggested an explosive reaction is a possible mechanism of alloy formation in the mechanical alloying, from his experimental result on Al-Ni. He reported an exothermic temperature spike has been observed during milling. Similar observations were reported by Shaffer[7,8] on some metal-metal oxide systems and by Lee et al.[9] on Mo-Si system. The authors[10,11] showed that the occurrence of the exothermic spike depends not on the total amount of impact energy given to feed powders but on the magnitude of impact energy given to the powders at every collisions.

Titanium silicides are expected to provide an excellent combination of high temperature strength and oxidation resistance at severe temperatures. This paper reports the formation of Ti_5Si_3 by combustion synthesis reaction during mechanical alloying. An exothermic temperature spike was observed when a powder with Ti-40at%Si composition was milled for 60hours, and aged for 48 hours and milled again for 32.5 hours. After this temperatutre spike, Ti_5Si_3 particles in a size range from several tens to several hundreds of nanometers were formed. On the other hand, no temperature spike was observed when milled continuously.

Experimental details

Elemental titanium(purity 99.9% and particle size less than 50μm) and silicon(purity 99.9% and particle size less than 74μm) powders were used as starting materials. A Ti/Si powder premix was put into a cylindrical mill container(70mm in inner diam. and 135mm in inner length) made of stainless steel(SUS-304) together with 25.4mm steel(SUJ-2) balls. Since the starting powders with compositions of Ti-13.67~80at%Si were milled. Ball charge was 60% of a maximum charge in the container and ball-to-powder weight ratio was 100:1. The container was sealed in an argon glove box. The vibration of a frequency of 25 Hz with an amplitude of 2.5mm was given to the mill container. The milling experiments were done on two different time-schedules. One group was milled continuously and the other was milled intermittently, that is, the powder was entirely taken out of the container and was observed by SEM. During milling, temperature of the milling container was measured by a thermocouple attached to the container surface. Cross sectional microstructure of the milled powders was observed by SEM on polished and etched sections of the milled powder particles mounted in an epoxyresin. Compositional distribution in the particles were inspected by EDAX using the polished and etched sections. X-ray diffraction analysis(XRD) was performed on the milled powders to monitor the phase transformation. Thermal behavior of the Ti-40at%Si composition powder was measured after various milling times by means of a differential thermal analysis(DTA).

Conclusions

The morphological evolution, amorphization and the formation process of titanium silicide particles during vibratory ball milling of the premixed Ti and Si powders were investigated. The exothermic temperature spike was only observed for the powder of Ti-40at%Si composition milled intermittently. After the temperature spike, the X-ray diffraction pattern and SEM show the formation of crystalline Ti_5Si_3 fine particles from the amorphous particles. On the other hand, no temperature spike was observed in the continuous milling and Ti-38atS%Si composition milled intermittently. In these cases, Ti_5Si_3 crystalline powder particles were formed gradually. The exothermic temperature spike is believed to indicate the combustion synthesis occurred during milling. The glass forming range of the mechanically alloyed Ti/Si is estimated roughly from 20 to 60at%Si under the present experimental condition.

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