Milling behaviour of nano-sized Sn powder prepared by electrical explosion method

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1. Introduction

Recently, Sn based material has been investigated as anode material for lithium ion battery due to high initial capacity for lithium ion. During cycle test, discharge capacity decreased with cycle because Sn powder expands and cracks. One of the most promising method for improvement of cycle-ability is to use nano-sized powder. Electrical explosion method is useful for making nano-sized metal powder. Milling process will be necessary for controlling the size and shape of nano-sized Sn powder.

In this process, SPEX milling behaviour of nano-sized Sn powder prepared by electrical explosion method was studied.

2. Experimental procedures

Nano-sized Sn powder was prepared by pulsed wire evaporation method. The Sn powder and carbon black were charged in jar (inner diameter 38mm, inner height 57mm) made of tungsten carbide based material. Two balls with diameter 1/2" and four balls with diameter 1/4" were also put into the milling jar filled with hexane and raw materials. The input ratio of Sn and carbon is 89.5 wt.%:10.5 wt.%. The milling time was varied with 10 min., 1 hr, 2 hrs, and 4 hrs, respectively. The milled powders were dried and the shape and size were observed by FE-SEM.

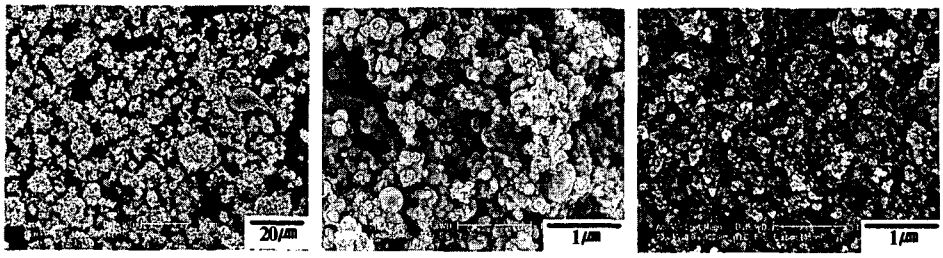
3. Experimental results

Fig. 1. shows the FE-SEM micrographs of Sn powder prepared by electrical explosion method. The sizes of agglomerated powders and individual particles were about 5 ㎛~20 ㎛ and 10 nm ~ 500 nm, respectively. The carbon used in this study also has size below 50 nm as shown in Fig. 1. (c).

The X-ray diffraction pattern of Sn powder shows Sn peaks and weak SnO peaks. Fig. 2. shows the shapes and sizes of milled Sn-C powder. After milling for 10 min., microstructure of Sn powders shows some large agglomerates consisting of very fine particles. The Sn powders were milled into flat shapes because Sn powder are easily plastic deformed by repeated impact force of balls. Especially, the heat generated by high energy milling may be contributed to agglomeration. After milling for 1 hrs, the sizes of agglomerates decreased to about below 100 ㎛. The large agglomerates were fragmented by impact of balls during milling for 4 hrs as shown in Fig. 2. (c). The sizes of agglomerates were about below 40 ㎛. As shown in a higher magnification of Fig. 2 (c), there are many nano-sized particles in agglomerates.

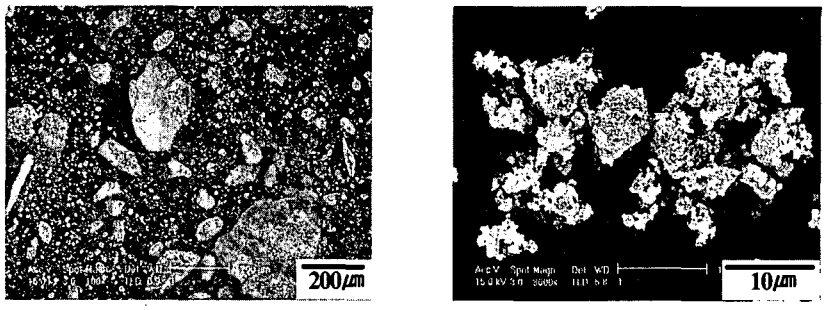
3. Conclusions

Nano-sized Sn powders were plastic-deformed and agglomerated by impact force of balls and heat generated during the SPEX milling. The agglomerated powder size decreased to below 40 ㎛ by milling for 4 hrs. The agglomerated Sn powder also consisted of many nano-sized particles.



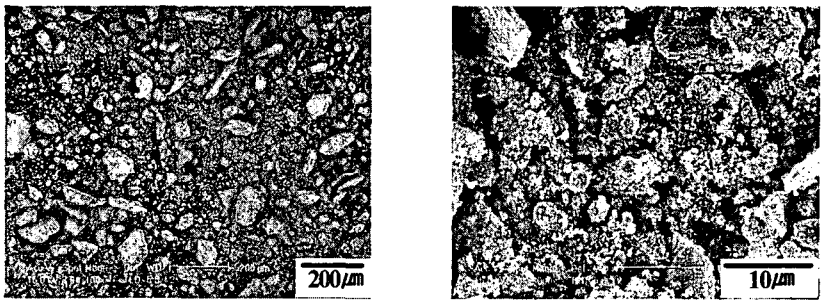
(a) Sn powder, ×1,000. (b) Sn powder, ×30,000. (c) Carbon powder, ×30,000

Fig. 1. Field Emission SEM micrographs of (a), (b) Sn powder and (b) carbon powder, respectively.



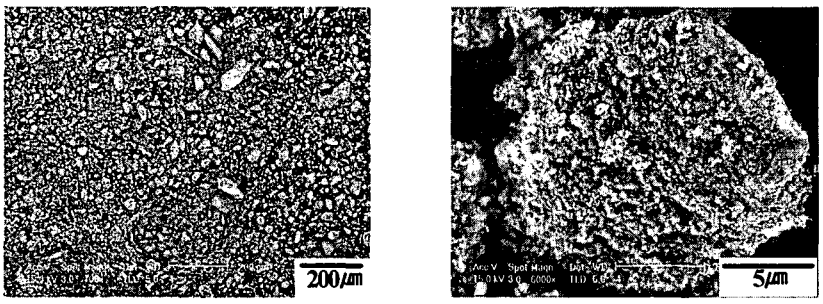
×100 ×3,000

(a) 10 min.



×100 ×3,000

(b) 1 hours



×100 ×6,000

(c) 4 hours

Fig. 2. Field Emission SEM micrographs of Sn powder with SPEX milling time, (a) 10 min., (b) 1 hour and (c) 4 hours, respectively.