Preparation and Characterization of Monosized Germanium Particles by Pulsated Orifice Ejection Method

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Abstract

Monosized germanium micro particles are prepared by a newly developed Pulsated Orifice Ejection Method. The obtained particles are categorized into two kinds of the microstructures as refined and coarse ones. The morphological difference is estimated to be determined by the undercooling level during nucleation. Actually, the increase in the temperature of the melt was effective in coarsening the microstructure, because the temperature of the melt intensely relates to the undercooling level. The transition temperature of coarse and refined microstructures is found to be 1300-1350K. Furthermore, a triggered nucleation could improve the crystallinity of the particles in the short separation.

Keywords : Pulsated Orifice Ejection Method, germanium particles, crystallinity, triggered nucleation

1. Introduction

Recently, spherical semiconductor technology has been received attracted instead of the conventional flat wafer in the electronic, optical and energy fields [1]. Since a spherical wafer has about three times utilized surface area of a conventional flat wafer, further integration of ICs is expected. Moreover, a spherical face provides a high freedom of device design on account of symmetry and no edge [2]. Actually, Ball ICs, spherical solar cells and 3D-accelerometers have been prototyped from semiconductor particles [3]. Although most of the industrial processes for the spherical semiconductor devices were devised, there is no promising process for the starting semiconductor particles. This is because the semiconductor particles are demanded mono-dispersity, high crystallinity, high purity in addition to mass-productivity. So far, we have succeeded in preparing monosized metal particles by our newly developed Pulsated Orifice Ejection Method (POEM) without sieving process [4, 5]. Accordingly, the POEM was expected to obtain the semiconductor micro particles at a high rate. Furthermore for improving the crystallinity of the particles, the influence of the temperature of the melt is investigated and a forced nucleation using trigger plate is attempted at a lower undercooling.

2. Experimental Procedures

Fig. 1 shows the schematic diagrama of a pulsated orifice ejection method. The raw materials was a germanium template (>40 cm, TDY Co. Ltd., Japan). The particles are prepared accorging to the following procedures. The starting material is placed in the tundish and heated up to above the melting point by an induction heater. The molten germanium is then ejected from the oscillation of the cylinder-rod which is driven by a piezoelectric actuator. The spherical germanium droplet is formed during flowing out of the melt from the orifice and solidified during falling in the Ar gas atmosphere.

The temperature of the melt was varied from 1253 to 1473K. Meanwhile, a trigger plate was equipped crossing

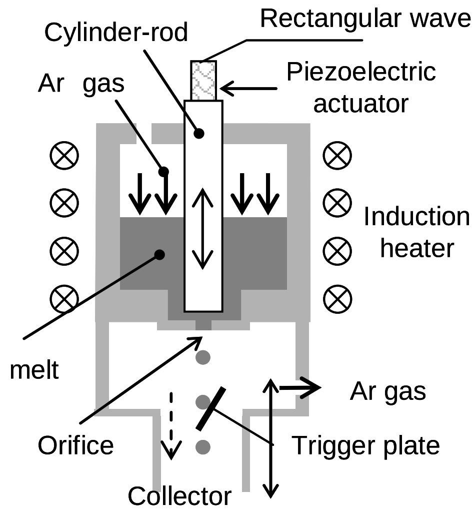

Fig. 1. Schematic illustration of POEM.

the falling trajectory of dro plet in the drop-tube for the forced nucleation. The obtained particles are observed by a scanning electron microscopy (SEM). The cross sectional observation of the obtained particles is conducted by FE-SEM equipped with an electron backscattering pattern (EBSP) as well as an orientation imaging microscopy (OIM).

3. Results and discussion

We have succeeded in preparation of monosized germanium particles by POEM. The diameter of particles is changed from 100 to 500um with optimizing the orifice diameter. The obtained particles are categorized into two kinds of microstructures as shown in Fig. 2. The particle in

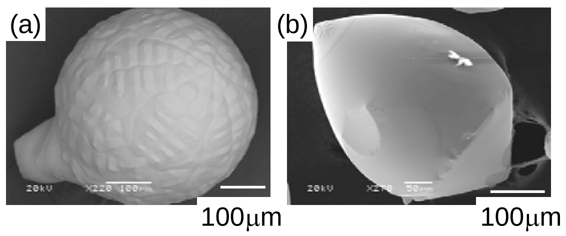

Fig. 2. SEM micrographs of typical Ge particles prepared by POEM.

(a) has nearly spherical shape with a protrusion and roughened surface. The protrusion is identified to be the latest solidification area, because the volume of semiconductor materials expands during solidification. Moreover the opposite pole of the protrusion is supposed to be a nucleation site, because the six-fold symmetric dendrites are formed. On the other hand, the particle (b), called "lateral particle" presents a tear shape with smooth surface. Moreover, the particle (b) presents the striation around the tip. Therefore, the particle of (b) is formed by lateral growth. Moreover the results of the crystallographical analysis clarify that the lateral particle has high crystallinity as compared with dendrite particle.

The lateral particles ratio of the obtained particles is counted as the function of the temperature of the melt. When the temperature of the melt is under 1300K, the obtained particles are almost the lateral ones. With increasing in the temperature of the melt, the lateral particles ratio is gradually decreased. The transition temperature of the melt from the lateral to dendritic is evaluated to be approximately 1300-1350K. This result demonstrates that the temperature of the melt is an important factor to form the high crystallinity particles.

Table 1. The repationship between the lateral particles ratio and contact position. The diameter of all particles is 200 μm

Contact position [mm]	30	50	-
Lateral paticles ratio [%]	75	10	0

To induce a nucleation at a lower undercooling, the triggered nucleation experiment is carried out. The relationship between the lateral particles ratio and the contact position is shown in Table 1.

Other process parameters are fixed in this experiment. The lateral particles ratio is obviously changed within the contact point between 30 and 50mm. Therefore, the triggered nucleation has a potential to control nucleation period.

4. Summary

Monosized spherical germanium particles were prepared by the pulsated orifice ejection method. The particles were morphologically categorized into two kinds of the microstructures: the dendritic particles composed of many grains and the lateral particles composed of a few grains. This definitive difference in crystallinity was determined by the undercooling during nucleation. Therefore, the transition temperature of the melt from the lateral particles to the dendritic ones was evaluated to be approximetry 1300-1350K. With employing the triggered nucleation, the lateral particles ratio was obviously increased in the contact position ranged from 30 to 50mm. Therefore, the triggered nucleation could improve the crystallinity of the particles in the short separation.

5. References

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