Preparation of Fine Particles by Gas Evaporation Technique with Electron Beam Heating

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The electron beam heating was applied to the gas evaporation technique to produce fine metallic particles. The advantages of the present method are that it works at very low pressures where the plasma flame heating does not work, and that it is free of the crucible damage which is serious in the case of the tungsten heater. It was found that copper particles of 50 mg were produced per minute in the argon gas of 0.5 Torr with an electron beam of 50 kV at 5 mA.

§1. Introduction

The method of preparing fine metal particles by the gas evaporation technique was developed by Kimoto *et al.*[1] in 1963. They used tungsten heater as the heating agent. However, the heater often reacts with the metal and burns out. The gas evaporation technique using a plasma jet flame as the heating agent has been developed by Wada[2] in 1969. This method also has a limitation that it cannot be used in low pressure atmosphere.

This paper describes a preliminary result of the technique to use an electron beam as the heating agent. It was shown that the copper particles of 50 mg per minute were produced in 0.5 Torr argon with an electron beam of 50 kV and 5 mA. The particle size was similar to the case of evaporation by a tungsten heater.[3]

§2. Apparatus

The apparatus used in the present experiment is schematically shown in Fig. 1. It consists of an electron gun chamber C_1, a middle chamber C_2 and an evaporation chamber C_3. The chamber C_3 is a cylinder with the diameter of 40 cm and 50 cm long. The chambers C_1 and C_2 are connected with a tube T_1 with the diameter of 5 mm and the length of 80 mm. Similarly, the chambers C_2 and C_3 are connected with a tube T_2 with the diameter of 4 mm and the length of 50 mm. The chamber C_3 has a hearth which is placed 10 cm below the bottom of the tube T_2. For Cu, Ag and Zn, a carbon crucible is used as the hearth and for metals reacted with the carbon crucible, such as Fe, Co and Ni, an alumina-coated carbon crucible. For the observation of the particles by electron microscope, specimen grids with evaporated carbon films are placed 15 cm obliquely above the hearth. Chambers C_1, C_2 and C_3 were evacuated by a diffusion pump (100 l/sec), a rotary pump (300 l/min) and a diffusion pump (300 l/sec), respectively.

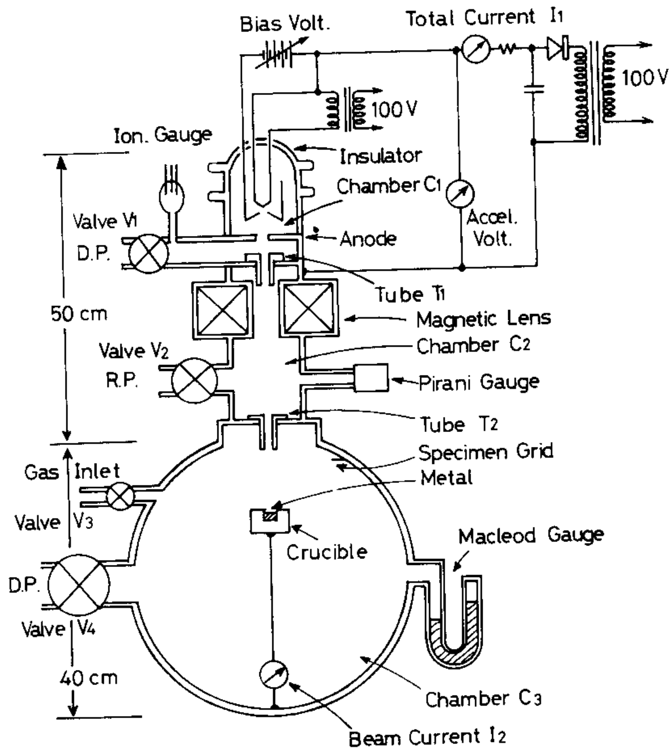

Fig. 1. Schematic diagram of the evaporation apparatus with an electron beam.

§3. Experimental Results

3.1 *Gas pressure P_3 of C_3 and accelerating voltage*

After evacuating chambers C_1, C_2 and C_3, valve V_4 was closed. A certain amount of argon gas was introduced into C_3 through the gas inlet and the pressures P_1, P_2 and P_3 of chambers C_1, C_2 and C_3, respectively, were measured. When the pressure P_3 was one Torr, those of P_1 and P_2 were 10^{-3} Torr and 10^{-1} Torr, respectively. Pressure P_3 decreased gradually, since the gas was evacuated through the tube T_2. The rate of the decrease was less than one per cent per minute. When pressure P_3 was more than one Torr, discharges took place in chamber C_1 at accelerating voltages over about 50 kV. It was found that the discharge was caused not only by the increase of the gas pressure but also by a very small amount of particles which had been introduced into the chamber C_1 and deposited on the insulator.

3.2 *Electron beam in the gas*

The electron beam in the gas became visible when the pressure P_3 was more than 0.01 Torr. It could be focused on the hearth by a magnetic lens at pressures below 0.1 Torr. It could not be focused well above 0.1 Torr. The beam current was divided into the current I_2 and I_3 as shown in Fig. 2. The former reached the hearth of diameter 6 mm and the latter was the part scattered outside the hearth. Fig. 3 shows that the current I_2 decreases due to the scattering in C_3 at pressures P_3 more than 0.1 Torr. The decrease of I_2+I_3 at the pressure P_3 more than one Torr suggests that the electron beam is already scattered in C_2. When the metal in the hearth began to evaporate, the currents I_2 and I_2+I_3 decreased to I_2' and $I_2'+I_3'$, respectively. It is understood that the electron beam is scattered by the metal vapour. When the carbon crucible was coated with an almina film, the surrounding gas of the crucible was slightly glowed by the beam. Therefore, the current I_2 seems to be carried by the gas ion.

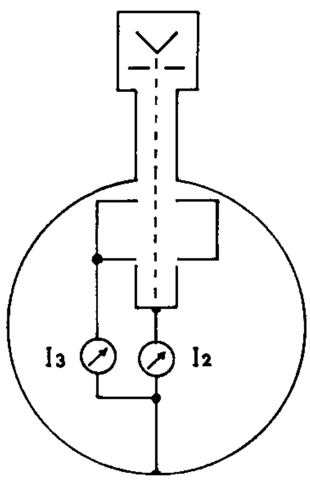

Fig. 2. Measurement of beam current.

3.3 *Evaporation of metal*

Metals evaporated were Bi, Sn, Ag, Mn, Cu, Mg, Fe, Fe-Co, Ni, Al and Zn. The amount of evaporated metal was estimated from the difference of the weight before and after the evaporation. For example, the weight of copper evaporated in a minute in the various gas pressures P_3 is shown in Fig. 4(a).

3.4 *Collected particles*

The particles were deposited like soot on the wall over the hearth. The amount of collected particles was about 0.1 gram for low melting point metals and 0.01 gram for high melting point ones per minute. The weight of the collected particles for copper is shown in Fig. 4(b), as an example. At pressures less than 0.1 Torr, a vacuum deposition film was obtained rather than the particles, Electron micrographs of particles were similar to the case of evaporation by a tungsten heater[3]. For example, the necklace arrangement is seen for Fe-Co alloy and the hexagonal habit for Zn.

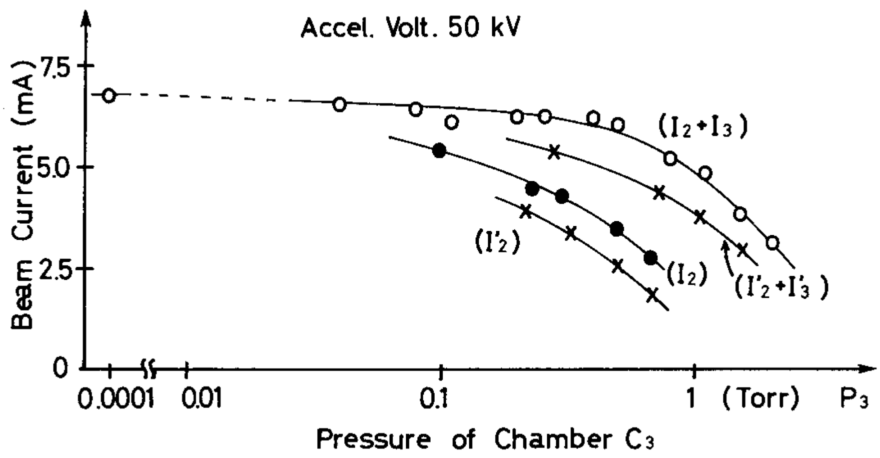

Fig. 3. Variation of the beam current with pressure P_3.

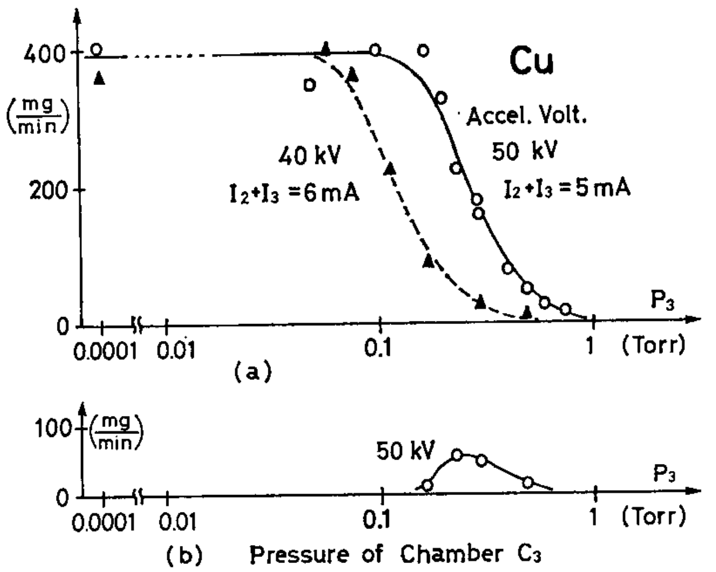

Fig. 4. Weight of evaporated metal (a) and that of collected particles (b).

§4. Discussion

It was shown that the particles was obtained with an electron beam as the heating agent. With the present apparatus, the gas pressure at which particles could be produced was lower than one Torr. However, it can be made higher with an increased accelerating voltage (Fig. 4).

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References

1) K. Kimoto, Y. Kamiya, M. Nonoyama and R. Uyeda: Japan. J. appl. Phys. **2** (1963) 702.
2) N. Wada: Japan. J. appl. Phys. **8** (1969) 551.
3) K. Kimoto and I. Nishida: Japan. J. appl. Phys. **6** (1967) 1047.