Development of Manufacturing Process of Tantalum Powder for the Capacitor

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This study examined the correlation of various operational factors including reaction temperature and the quantity of reductant and diluent with the characteristics of powder using K_2TaF_7 as feed materials, Na as a reductant and KCl/KF as a diluent. In addition, this study examined various types of after-treatment that affect the high purification of powder. Lastly, to control the particle size and shape, it developed equipment that can provide a feed material and a reductant at a fixed quantity and evaluated the characteristics of powder manufactured according to the manufacturing process stated above.

Major results of the experiment obtained from operational factors mentioned above are as follows.

1) The chemical composition of most of a small quantity of impurities in recovered powder after water washing was within the tolerance required for commercial tantalum powder. On the other hand, the quantities of Fe, Cr, Ni, Ca, K, Na, O were larger than commercial tantalum powder. In particular, the quantities of heavy metal impurities such as Fe, Cr, and Ni increased as the temperature in the reaction container went high when the quantity of reductant was large, the reaction temperature was high, and the quantity of diluent was small.
2) In case of a batch type process in which feed material, reductant, and diluent were charged at the same time, the reaction rate, recovery rate and particle size were high when the quantity of reductant was large, the reaction temperature was high and the quantity of diluent was small. In addition, when comparing several characteristics including the purity of recovered powder, the recovery rate and particle size, the optimal quantity of reductant added was +5wt% of the stoichiometric amount, the optimal reaction temperature was 850C, and the optimal ratio of feed material to diluent was 1:1.
3) A significant amount of impurities contained in recovered powder was removed in various conditions of acid washing. In particular, 20% (HCl + HNO_3) was effective in removing heavy metal impurities such as Fe, Cr, and Ni, 8% H_2SO_4 + 8% $Al_2(SO_4)_3$ in removing fluorides such as K and F from non-reactive feed material, and 2% H_2O_2 + 1% HF in removing oxides that formed during reaction.
4) Significant amounts of oxygen and part of light metal impurities could be removed through deoxidation and heat treatment process. On the other hand, because it is difficult to remove completely heavy metal impurities such as Fe, Cr, and Ni through

acid washing or heat treatment process if their contents are too high, it is considered desirable to inhibit these impurities from being mixed during the reduction process as much as possible.

5) When the external supply system was applied instead of the batch type process that charges feed material, reductant and diluent at the same time, it was possible to induce regular reduction reaction between feed material and reductant, which increased the recovery rate and reduced the mixture of impurities.
6) In particular, the application of the external supply system enabled the control of reaction temperature and reaction speed according to the feeding rate of feed material during reduced reaction, and resultantly it enabled the manufacturing of granular-shaped powder with a regular granularity of 1~3μm and purity of 99.5%.
7) Their electric properties were measured by an external test agent. According to the result, leakage current(L.C) and dielectric dissipation(tanδ) were low with high reliability and capacitance was 23,000CV.