RECOVERY OF TANTALUM FROM CAPACITOR WITH SOLVOTHERMAL TREATMENT

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Abstract

Liquefaction of resin in the tantalum capacitors were challenged in sub-critical fluids with a batch-type stainless steel (SUS316) reactor for perfect recovery of high purity tantalum and also efficient conversion to valuable chemicals When 1-Methyl-2-pyrrolidone (NMP) was used as a solvent for the capacitor treatment, we found that resin was able to be solubilized and separated from the capacitor at 300 °C for 6 h in the presence of K_3PO_4 catalyst. In contrast, in the case of benzyl alcohol (BZA) solvent, the solubilization rate of resin was lower than that in case of NMP at 300 oC for 4 hrs with catalyst. We also found that resin solubilization of resin without any catalyst became faster than that in the presence of catalyst.

Keywords: *Subcritical fluid, benzyl alcohol, Capacitor, Tantalum, Solvothermal.*

1. Introduction

Tantalum capacitor is a small-sized and has large capacity, and is indispensable for electronic equipment. The demand increases year by year. In addition, there are quite large number of non-standard products (scrap) generated during the capacitor manufacturing process and used tantalum capacitors. If both the efficient conversion of resin to valuable chemicals and perfect recovery of tantalum can be realized, the technique can be utilized as a novel and effective usage method of precious petroleum resources. So far, a new technique with oxidative degradation of tantalum capacitors at high temperatures has been proposed. However, there was room for improvement in the points that reactions have to be carried out at high temperatures like 1,200 °C and that this technique required leaching processing this technique.

In this study, we aimed to develop a novel technique for complete recovery of tantalum from capacitors in high temperature and high-pressure solvents (1-Methyl-2-pyrrolidone (NMP) and benzyl alcohol (BZA)). Also, we investigated possibility that polymer resin in the capacitors was able to be depolymerized or liquefied to valuable chemicals. Especially, in case of BZA use, we challenged to explore optimum conditions where tantalum was successfully able to recover from the capacitors by this technique. If sub-critical BZA is found to be applicable to chemical recycling, the present chemical recycling processes using NMP solvents will be alternate to more environmentally benign ones.

2. Materials and Methods

Tantalum capacitors were used as starting materials. NMP, which is commonly used as a solvent, and BZA were used as reactive solvents in this study. In case of catalytic reaction experiments, K_3PO_4 catalyst (Wako Pure Chemicals, Osaka) was also used.

Reactions of tantalum capacitors were carried out with a batch-type stainless steel (SUS316) reactor whose inner volume was 8.8 mL. About 3 g of tantalum capacitors, 3 mL of NMP (or BZA), and K_3PO_4 catalyst (final concentration: 0.33 mol/L) were loaded into the reactor. After sealed, the reactor was put into an electric furnace (see Fig. 2) which had already preheated at desired reaction temperatures (275 – 300 °C) and reactions of tantalum capacitor were initiated. After 1 – 6 h, it was quenched rapidly by iced water bath to terminate further reaction.

Fig. 1 A batch-type reactor used in this study.

Fig. 2 Electric furnace for solvothermal treatment of tantalum capacitor.

Products were filtrated and liquid and solid portions were obtained. The solid portion was fractionated by grounding by hand about 10 times and by using a wire sieve (16 mesh) with repeated water washings to remove polymer resin-based portion from solid one. After drying, the solid portion was weighed and then calculated liquefaction rate (X) and resin removal rate (X_R) by the following equations;

$$X\ (\text{wt\%}) = (1 - W/W_0) \times 100 \quad (1)$$
$$X_R\ (\text{wt\%}) = X/X_0 \times 100 \quad (2)$$

where W_0 is the dry weight of capacitors (g), W is the dry weight of solid residues (g), and X_0 is initial content of resin in the tantalum capacitor which has been determined in the preliminary experiment (in this case, X_0 = 43 wt%).
The solid residue obtained was also analysed by XRF, XPS and so on to understand the elemental composition.

3. Results and Discussion

At first, in order to understand optimum treatment time for complete capacitor fractionation between tantalum and polymer resins, liquefaction experiments were conducted at various treatment times at a constant temperature of 300 °C in the presence of K_3PO_4 catalyst. Table 1 summarizes a time course of liquefaction rate (X) and resin removal rate (X_R) of tantalum capacitor. The X value increased with treatment time and reached 41 wt% for 4 hrs, especially for more than 6 hrs the resin was perfectly separated from capacitors. From this result, we found that NMP at the subcritical state (300 °C) was able to solubilize resin in the capacitor and to recover tantalum at identical condition.

Table 1. Time course of liquefaction rate (X) and resin removal rate (X_R) of tantalum capacitor in sub-critical NMP at 300 °C with K_3PO_4 catalyst.

Time / h	2	4	6	8
X / wt%	15	41	43	43
X_R / wt%	36	95	100	100

Next, BZA solvent was employed to the treatment of tantalum capacitor. Fig. 3 shows the result. In case of BZA solvent, the X value was 7.2 wt% at 300 °C for 4 hrs in the presence of K_3PO_4 catalyst, which was lower than that in case of NMP solvent.

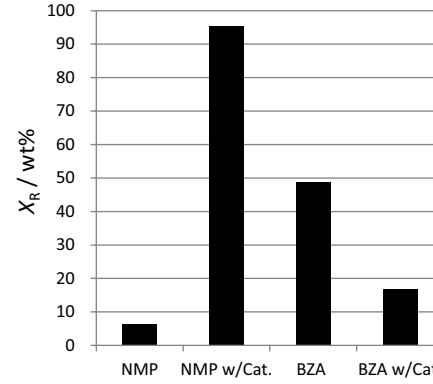

Fig. 3 Comparison of the X_R values in sub-critical NMP and BZA at 300 °C for 4 hrs.

This result clearly indicates that the operating condition should be optimized in the case of BZA solvent. Also, we found from the result that the liquefaction rate and resin removal rate in the absence of K_3PO_4 catalyst became higher than those in the presence of the catalyst. Although the reaction mechanism for resin depolymerization in sub-critical BZA is now unclear, but will be provided at the conference.

Acknowledgement

This work was performed by the support of Kumamoto University Global COE (Centers of Excellence) program “Global Initiative Center for Pulsed Power Engineering”.