

# Preliminary Information of Laboratorial Tantalum Recovery and Considerations for a Potential Solution for Conflict Mineral and Wildlife Conservation

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## Abstract

Tantalum is a rarely occurring transition metal which is chiefly used to manufacture capacitors for electronic appliances such as mobile phones. The demand for tantalum is expected to show an annual growth rate of about 5%; however, tantalum produced in the Democratic Republic of Congo is considered a conflict mineral due to illegal mining which is accelerating the extinction of lowland gorillas. Although tantalum recycling is desirable, the recycling technology has been poorly developed. Phase separation was applied to recover tantalum. It was expected that a copper phase could absorb tantalum, but tantalum was slightly transferred into an iron-rich phase in the Cu-Fe-C system. When ferric oxide was applied as an auxiliary, the tantalum was concentrated as precipitate on the surface of the iron-rich phase. In terms of practical application, a theoretical prediction suggests that the addition of a reductant will make it possible to recover tantalum as a high-purity powder. Since conflict, illicit exploration and hunting (bush-meat trade) have caused a decline in the population of wild animals, it is considered that a feasible tantalum recycling system will be helpful in solving such problems.

**Keywords:** African gorilla, conflict mineral, Dodd-Frank Act, mobile phone, phase separation, tantalum

## 1. Introduction

Tantalum (Ta) is a rarely occurring transition metal with high corrosion resistance at normal temperature. It is chiefly used to manufacture capacitors for electronic appliances such as mobile phones, laptop computers, and portable music players (Larsen, 2002). Tantalum demand has grown globally in the last two decades, and annual consumption reached about 2,600 tons Ta<sub>2</sub>O<sub>5</sub> in 2001 because a large volume of tantalum was consumed to produce capacitors for electronic equipment (Roskill Information Services [RIS], 2005). Demand is expected to show an annual growth rate of ~5% (RIS, 2005).

About 18% of tantalum is used to manufacture tantalum chemicals, 17% is used to produce metallurgical grade tantalum powders, and a further 24% is used in the production of capacitor-grade powder (Schwele, 2010). According to other data (Shaw et al., 2011), capacitor-grade powder is estimated at 48%. Tantalum is mostly recycled from waste material and scrap that are generated during the manufacture of tantalum-related electronic components (Cunningham, 2004). The global recycling rate is about 20% (Cunningham, 2004; Ad-hoc Working Group of Raw Materials Supply [AWGRMS], 2010; Japan Oil, Gas and Metals National Corporation [JOGMEC], 2011); however, the recycling of tantalum from tantalum-based capacitors is difficult (Shaw et al., 2011). This difficulty may originate in the coexistence of tantalum with several materials - a capacitor consists of a metal terminal (Cu, Fe or Ni), a packing made of epoxy resin, and a tantalum electrode (Matsuoka et al., 2004); a schematic of a typical Ta capacitor is shown in Figure 1. It is reported that the current technology for tantalum recycling has been poorly developed (Shaw et al., 2011; Cunningham, 1998). As a large amount of energy is required to isolate tantalum from ore (Matsuoka et al., 2004), it is considered that the recycling of tantalum may help to increase resource efficiency in production and consumption.

Although tantalum production is comparatively widespread, Rwanda and the Democratic Republic of Congo

predominantly account for over 60% of global production; additional production comes from Brazil with minor amounts also produced in Malaysia and Canada (Shaw et al., 2011). Conflict minerals are exploited by a group of armed men to finance their struggle and militarization, and global attention is focused on the Democratic Republic of Congo where insurrection has lasted fifteen years (Business for Social Responsibility [BSR], 2011). The U.S. Securities and Exchange Commission published final rules for conflict minerals (the so-called Dodd-Frank Act) in August 2012 (Hileman, 2012). The published act requires companies that make products containing tantalum, tin, tungsten or gold to disclose whether their supply comes from or near the Democratic Republic of Congo. It is necessary to provide transparency regarding the supply chain to comply with this act. It is considered that the recycling of tantalum may help to solve the conflict mineral issue because the implementation of recycling will lead to a shift in the sourcing of the resource from import to domestic.

For the above-mentioned reasons, a laboratorial study was performed to find a way to separate tantalum from a mixture (capacitors in particular). Based on the obtained results, preliminary information on tantalum recycling is reported in the present paper, and considerations are given from the viewpoints of practical application, environmental aspects and economics.

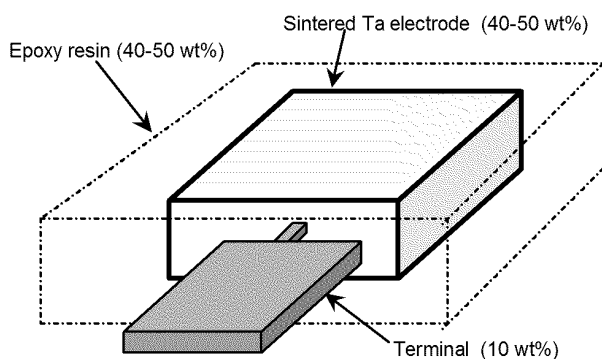


Figure 1. Schematic of a typical Ta capacitor showing material composition (Mineta & Okabe, 2005)

## 2. Materials and Methods

The binary melt of iron and copper is a miscible composition at high temperatures (Massalski & Okamoto, 2000), and the addition of carbon or phosphorus to the binary melt leads to a phase separation into liquid iron-rich and copper-rich phases (Hino, Washizu, & Nagasaka, 1998). The copper phase can generally absorb some precious metals such as gold and palladium (Yamaguchi et al., 2006).

As mentioned in the previous section, a capacitor is made of two inorganic components - a metal terminal (e.g. Fe and Cu) and a tantalum electrode. Assuming that tantalum is to be separated from these components, the distributions of tantalum by phase separation in a Cu-Fe-C system were studied using the following procedures in order to acquire basic knowledge for an industrial-scale application.

### 2.1 Carbon-Saturated Iron

Carbon-saturated iron was prepared by melting a mixture of 95.1 g Fe and 4.5 g C in a graphite crucible in an electric furnace (double spiral-shape SiC type) with an argon atmosphere at 1400 °C for 3 hours.

### 2.2 Sample Preparation

Ten grams of carbon-saturated iron and an equal amount of copper were mixed with, respectively, 0.05 g, 0.10 g and 0.20 g of tantalum, each in a graphite crucible having a height of 80 mm and an inside diameter of 11 mm.

Other samples were also prepared for a supplementary test in order to study the effect of iron oxides; i.e. 2.0 g of ferric oxide ( $\text{Fe}_2\text{O}_3$ ) was added to a mixture of 94.2 g carbon-saturated iron, 4.8 g copper and 1.0 g tantalum in an alumina crucible. All the crucibles except the alumina crucible were kept in the furnace with an argon atmosphere at 1250 °C for 15 hours, and only the alumina crucible was kept in the furnace for 1 hour.

### 2.3 Sample Collection

After all the crucibles were cooled to room temperature, both phases in the graphite crucibles were cut out for chemical analysis, and extraneous matter on the upper phase of the alumina crucible was picked up for observation.

## 2.4 Analysis and Observation

The phases collected for chemical analysis were sliced into ~0.1 g pieces with a sample cutting machine, and these pieces were weighed. Each sliced piece was put into a separate teflon beaker together with 5 ml aqua regia, and the beakers were kept on a hot plate. After 10 minutes, 9 ml sulfuric acid, 2 ml phosphoric acid and 3 ml hydrofluoric acid were added, and then these beakers were again heated for 1 hour. The solutions obtained in the beakers were cooled to room temperature and filtrated for qualitative analysis using inductively coupled plasma atomic emission spectrometry.

The extraneous matter scraped from the upper phase in the alumina crucible was directly investigated with a transmission electron microscope (TEM) and an energy dispersive X-ray analyzer (EDX).

## 3. Results

This section presents the results obtained from the distribution experiment, the effect test of iron oxides and TEM/EDX observation as follows.

### 3.1 Tantalum Distribution by Phase

In the graphite crucible, two phases were observed, one each in the upper part and the bottom part, which correspond to the iron-rich alloy and copper-rich alloy respectively. Figure 2 shows a cross section of the solidified sample obtained in this experiment for a Cu-Fe-C system at 1250 °C.

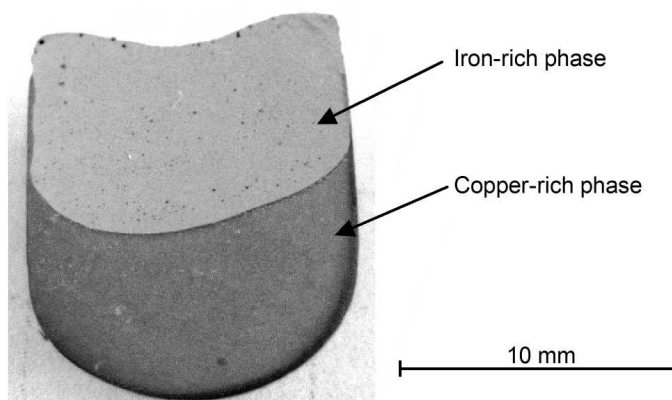


Figure 2. View of two phases in the Cu-Fe-C system at 1250 °C

Table 1. Material distribution by phase, with the input amount of tantalum as a parameter

Ta input amount (g)	Iron-rich phase		Copper-rich phase	
	Ta (wt %)	Cu (wt %)	Ta (wt %)	Fe (wt %)
0.05	0.33	4.58	0.01	3.88
0.10	0.49	4.86	0.02	3.81
0.20	1.01	3.81	0.01	3.71

It follows from Table 1 that the Ta content in the iron-rich phase is greater than that in the copper-rich phase. This table also indicates that the Ta content increases with an increase in the initial input of Ta in the iron-rich phase, but the Ta content is almost independent of the Ta input in the copper-rich phase.

### 3.2 Effect of Iron Oxides on Tantalum Distribution

As stated in section 2.2, ferric oxide ( $\text{Fe}_2\text{O}_3$ ) was added to the mixture of carbon-saturated iron, copper and tantalum in an alumina crucible. When this mixture was melted, the two phases did not clearly separate - i.e. the iron-rich phase mainly appeared; hence, samples were scraped from this iron phase. Table 2 summarizes the chemical compositions of the obtained samples. Tantalum was concentrated in the iron-rich phase without  $\text{Fe}_2\text{O}_3$  addition (Table 1), but it was not concentrated in the iron-rich phase with  $\text{Fe}_2\text{O}_3$  addition.

Table 2. Composition of iron-rich phase prepared by adding Fe<sub>2</sub>O<sub>3</sub>

Sampling position	Fe (wt %)	C (wt %)	Cu (wt %)	Ta (wt %)
Upper part	91.7	4.18	3.95	0.11
Middle part	91.3	4.18	4.19	0.12
Lower part	91.8	4.17	4.16	0.13

### 3.3 Observation by Electron Microscope Using Energy Dispersive X-ray Analyzer

Black powders, white powders and ashes came out in the iron-rich phase prepared by adding Fe<sub>2</sub>O<sub>3</sub>. Figure 3 shows the TEM micrographs of these precipitates, and arrows in this figure mark the areas analyzed by EDX linescan. Six elements including O, Cu, Fe and Ta were analyzed in the different areas on the samples, and the obtained results are summarized in Table 3. As compared with the Ta contents in the phase prepared by adding Fe<sub>2</sub>O<sub>3</sub> (Table 2), these precipitates contained large amounts of tantalum.

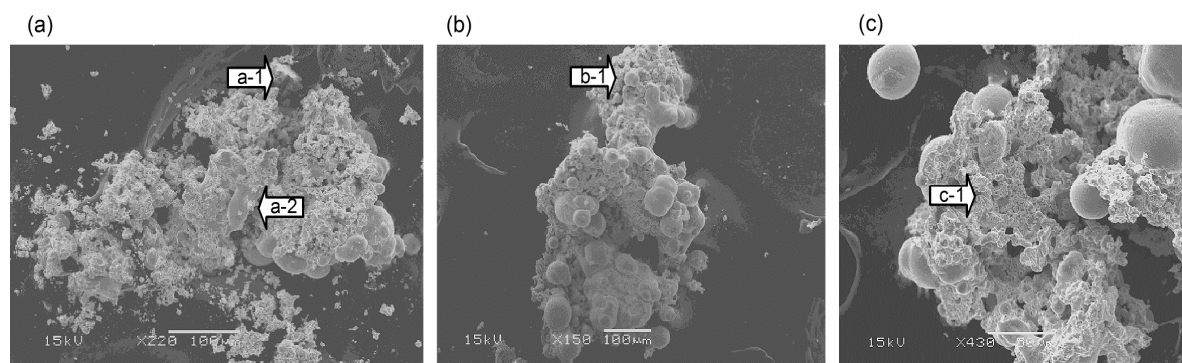


Figure 3. TEM micrographs of precipitate in iron-rich phase prepared by adding Fe<sub>2</sub>O<sub>3</sub>: (a) white precipitate, (b) black precipitate; and (c) ash-like precipitate. Arrow marks the EDX analysis area whose identification is represented in Table 3

Table 3. Composition of precipitates in the iron-rich phase prepared by adding Fe<sub>2</sub>O<sub>3</sub>

Analysis position	O (wt %)	Cu (wt %)	Al (wt %)	Si (wt %)	Fe (wt %)	Ta (wt %)
a-1	9.8	0.5	5.7	0.0	23.2	60.7
a-2	13.3	0.7	8.6	2.5	19.5	55.4
b-1	12.4	0.7	2.6	0.0	27.9	56.4
c-1	13.7	0.7	7.1	0.0	7.4	71.1

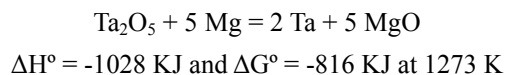
Positions of EDX analysis are referred to in Figure 3.

## 4. Consideration

A binary melt of iron and copper was not often obtained at the preparation step. As it is considered that the heating temperature and/or duration may cause such incomplete melting, a high-performance furnace and more heat-resistant cube will be required to effectively carry out a laboratorial test of phase separation for tantalum enrichment. The function of the oxide auxiliary is interpreted as follows: Tantalum was slightly transferred into an iron-rich phase in the Cu-Fe-C system (Table 1). When ferric oxide was applied as an auxiliary, the tantalum was concentrated as precipitate on the surface of the iron-rich phase (Table 3). This mechanism may be interpreted as follows: (i) first oxidation transfers Ta-Fe alloy into an Fe matrix phase; (ii) the oxygen affinity of Ta has to be sufficiently greater than that of Fe in order to oxidize a major portion of Ta; and (iii) the Ta atoms diffuse to the phase surface and form an external oxide layer.

Considering the wide applicability of tantalum, the recovery of tantalum as powder may be preferred. Tantalum oxide powder can be reduced to tantalum powder, so some reduction processes for tantalum oxides have already

been reported: for example, tantalum powder with capacitor grade can be produced by sodiothermic reduction that is generally called the Hunter Process (cf. Cho et al., 2006); tantalum oxide is reduced by calcium and this reductant is supplied to the oxide through molten  $\text{CaCl}_2$  (Baba et al., 2005); it is also proposed to produce tantalum from its oxide by aluminothermic reduction in a plasma reactor (Brito et al., 2008). Direct reduction processes such as calciothermic reduction and aluminothermic reduction have potential in future tantalum production (Okabe et al., 2003). When a new process is developed, its scalability and environmental issues should be considered. Magnesiothermic reduction is quite exothermic, and this overall reaction is summarized as follows (Barin, 1995):



When the reductant magnesium makes physical contact with  $\text{Ta}_2\text{O}_5$ , reductant impurities (e.g. N and C) are transported to the tantalum phase, and thus contaminate the metal product. To take measures against this contamination, magnesium vapor is usually applied for oxide reduction (Ono & Miyazaki, 1985) - that is, a metallothermic reduction in the vapor phase. This reduction is efficacious in controlling the reductant contamination, and tantalum powder can be obtained at 99% purity or more (Okabe et al., 2003). Since it is not easy to induce a homogeneous reaction on a large scale, it remains for a future study to examine how to scale up the process from the laboratory to a pilot plant.

The recovery of tantalum was studied on a laboratorial scale in order to obtain preliminary information, and this study indicates that tantalum recycling is technically possible. Practical application and its significance are discussed below with regard to tantalum recovery.

#### *4.1 Approach to Pre-Feasibility Study of Practical Application*

As stated in the introduction, the greatest end-use of tantalum is capacitor production for mobile phones, laptop computers, etc. The demand for tantalum capacitors has been increasing owing to the widespread use of the above-mentioned equipment. One reason is that these capacitors have a large capacity and high thermal stability compared to other capacitors (Matsuoka et al., 2004). It is therefore essential to consider whether tantalum can be recovered from end-of-life capacitors, and from the practical viewpoint this recovery should be proved by a pilot test. A full-scale feasibility study is still to be conducted, but a theoretical consideration is simply given as follows:

Since a tantalum capacitor consists of a packing made of epoxy resin, a metal terminal and a tantalum electrode (Figure 1), it is necessary to combine separation techniques in order to recover the tantalum. Firstly, a capacitor is heated in the air to thermally decompose the packing resin, and  $\text{SiO}_2$  residues are removed by sieving and water flushing (Matsuoka et al., 2004). The remains are basically composed of three components: a Cu terminal, Ta electrode, and Ta wire for linking the Ta electrode to the Cu terminal - i.e. these components are made from tantalum and copper. The obtained results suggest that the copper component melts in the copper-rich phase, and tantalum precipitates as oxide ( $\text{TaOx}$ ) on the surface of the iron-rich phase by means of phase separation in the Cu-Fe-C system prepared by adding  $\text{Fe}_2\text{O}_3$ . Thus, from the technical viewpoint it is considered that tantalum can be recovered as an oxide from an end-of-life capacitor; however, it remains for a future study to determine detailed parameters such as process operability, cost performance, engineering data leading to industrial plant design, etc.

#### *4.2 Economic and Social Viewpoints*

As stated in the introduction, tantalum production involves several issues: (i) international pacifism - global attention on conflict minerals is presently focused on the Democratic Republic of Congo, and the Dodd-Frank Act requires companies that make products containing tantalum and the like to disclose whether their supply comes from or near the Democratic Republic of Congo; (ii) resource localization - the Democratic Republic of Congo is a main producer of tantalum, and this country predominantly accounts for over 60% of global production; and (iii) demand enlargement - the demand for tantalum capacitors has been increasing owing to the widespread use of electronic devices such as mobile phones. Therefore, the recycling of tantalum should be discussed not only from the techno-economic viewpoint but also from the above-mentioned viewpoints because material recycling implies a choice to balance the demands of a growing economy with social and environmental needs.

Tantalum is commercially different from many other metals because it is not traded on any open exchange; buyer and seller bargain regarding contract prices, and these prices usually remain secret (Shaw et al., 2011). Trade information sometimes reports indicative prices derived from interviews with buyers and sellers. Tantalum is

traded in three forms, commanding different prices: tantalum ore concentrate was about US\$265/kg, tantalum oxides and salts about US\$530/kg, and capacitor-grade powder in excess of US\$660/kg in February 2011 (Reisman, 2011). These prices should not be simply compared with the recycling cost because tantalum is classified as a type of conflict mineral (cf. the introduction). Locals in mining communities are forced to take part in the illicit mining economy. Money earned from the sale of conflict minerals is used for personal profit and for further violent causes. Minerals are smuggled out of the conflict site through neighboring countries, and then shipped to smelters around the world for refinement. Once minerals are processed in this way, it is difficult to trace their origin. Conflict minerals easily make their way to consumers all over the world. As the imported portion of conflict minerals will be replaced by a domestic portion following the implementation of recycling, dependence on imports can at least be partially reduced. In other words, there is a possibility that the recycling of tantalum may contribute to not only price and supply stabilization in a consumer economy but also economic sanctions against illicit mining communities. Since it is difficult to trace the origin of tantalum through a transit and laundering point for conflict minerals, the recycling of tantalum from post-consumer capacitors must be helpful in clarifying the supply chain.

#### *4.3 Environmental Viewpoint*

Due to increasing environmental awareness, it is necessary to minimize the impacts of tantalum processing on the environment. Since the manufacture of capacitor-grade tantalum powder requires the consumption of alkali halide salts, sodium, magnesium and mineral acids (Ruch, 2010), various types of waste are released from the processing (Tantalum Story Board [TSB], 2010). The phase separation applied currently is a dry type of metallurgical process, so the waste release is less than that from conventional tantalum processing. The significance of tantalum recycling is discussed below from the viewpoint of nature conservation.

Columbite-tantalite - coltan for short - is extracted from niobium and tantalum; cf. the niobium-dominant mineral is columbite and the tantalum-dominant mineral is tantalite in coltan (Roberts et al., 1990). Price rises of coltan have led to wide-scale unregulated mining in the forests of Congo, the same forests that were home to 86 % of eastern lowland gorillas (Hall et al., 1998). The forests are being destroyed, and gorillas are killed and snared as "bush meat" to feed local miners and sell at market (Redmond, 2001). It is estimated that 90% of lowland gorillas may have been killed (Redmond, 2001). Fears are that without immediate action, they may be headed towards inevitable extinction.

As stated in the introduction, tantalum is mainly used to produce capacitors for electronic equipment. Forty-seven American zoos (e.g. San Diego Zoo) are directing their attention to the hidden connection between mobile phones and the decline of lowland gorillas (Lovgren, 2006); that is, according to a comment by an associate curator of San Diego Zoo, recycling old mobile phones is a way for people to do something simple that could reduce the need for additional coltan and help to protect the gorillas. From this viewpoint, if tantalum contained in mobile phones is effectively recovered, that might also help diminish the demand for illegal coltan mining, which could in fact help gorillas and other animals to live safely in their habitat.

### **5. Conclusion**

Even if regulations for controlling conflict minerals are strictly enforced, it is almost impossible to trace the origin of tantalum through a transit and laundering point. It stands to reason that the recycling of tantalum from post-consumer capacitors must be helpful in clarifying the supply chain and reducing illegal mining; in short, the issues surrounding tantalum are a good example of how recycling is closely linked with the need for handy electronic devices, the peace movement and nature conservation.

In spite of such importance, the current technology for tantalum recycling has been poorly developed. Tantalum is mostly recycled from waste material and scrap that are generated during the manufacture of tantalum-related electronic components; on the other hand, since a large amount of tantalum is contained in end-of-life capacitors, the recycling of tantalum from tantalum-based capacitors is highly desirable – i.e. there is a gap between the quantity recovered by the available technology and the abundance of the recycle source. Considering such gap, the present paper aimed to obtain preliminary information on tantalum recycling to compensate for the gap. The following points are established – (i) tantalum purity of over 70% from the described mode of recovery is possible, and (ii) a theoretical prediction suggests that tantalum powder with purity greater than 99% can be produced from end-of-life capacitors. Although it can be considered that the present study establishes the foundations for tantalum recycling, it remains for a future study to evaluate the techno-economic performance and design of an industrial process in detail.

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