Production of Tantalum powders using the Cambridge FFC Reduction Method

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Abstract

The paper will show the progress made from the discovery of the FFC reduction method at Cambridge University in 1997, with reference to Tantalum Capacitor Grade powder.
Metalysis will show the reduction from Ta_2O_5 to tantalum powder via an electro chemical reduction in molten salt. Lower environmental impact, when compared to the alternative reduction methods.

The presentation will include the chemical , physical and electrical properties of FFC capacitor grade powder. This will demonstrate the interesting tantalum powder morphology, produced from the FFC reduction method.

Introduction

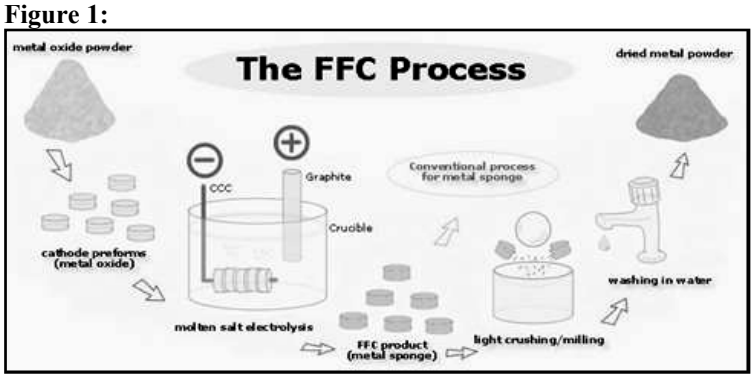
The Cambridge FFC process uses a molten salt as the electrolyte for the electrochemical reduction of a metal oxide to metal. The process fundamentals and chemistry have been described in a series of papers in the open and patent literature [1-3].
The metal oxide powder is contained in the cathode and directly converted into metal by electro-deoxidation. The oxygen ions carry the current across the cell and gas is evolved at the anode, leaving the pure metal at the cathode. For tantalum, the product can simply be processed to a powder. Figure 1 shows the basic FFC process.

When compared to the sodium reduction technique, the Cambridge FFC process is extremely elegant and has a low environmental impact. The process operates at low temperatures (in the range 750 – 1000^{o}C) and analysis has shown that it compares

very favourably to sodium reduction technique with regards to energy efficiency. The only by products of the Cambridge FFC process is comparatively small levels of carbon dioxide and carbon monoxide, along with the calcium chloride (salt). A program is in place to reuse the calcium chloride, thus reducing this environmental impact.

The Cambridge FFC process only removes oxygen from the Ta_2O_5. Because it is not a refining process great care must be taken in the material construction of the Metalysis FFC reduction cell and the purity of the tantalum oxide pellet. With post processing of the tantalum powder in various acids the tramp elements can be reduced to meet tantalum specifications.

Figure 1:



Metalysis FFC reduction chain

The Cambridge FFC process offers a potential to produce various grades of capacitor grade tantalum powder with very little post processing .The sodium reduction method involves a number of complex steps and the use of un-environmentally friendly reactants to complete the process. The Metalysis FFC process cuts a larger number of production steps out (figure 2) and has some advantages over the sodium reduction method for example:

- Avoids the forming of oxyfluorides.
- The morphology is generated in the Cambridge FFC reduction.
- By altering the process parameters, Metalysis can obtain a wide range of surface areas (BET of 0.1 to 10m^2/g).

<u>Sodium reduction</u>

K_2TaF_7+5 Na → Ta+5NaF+2 KF

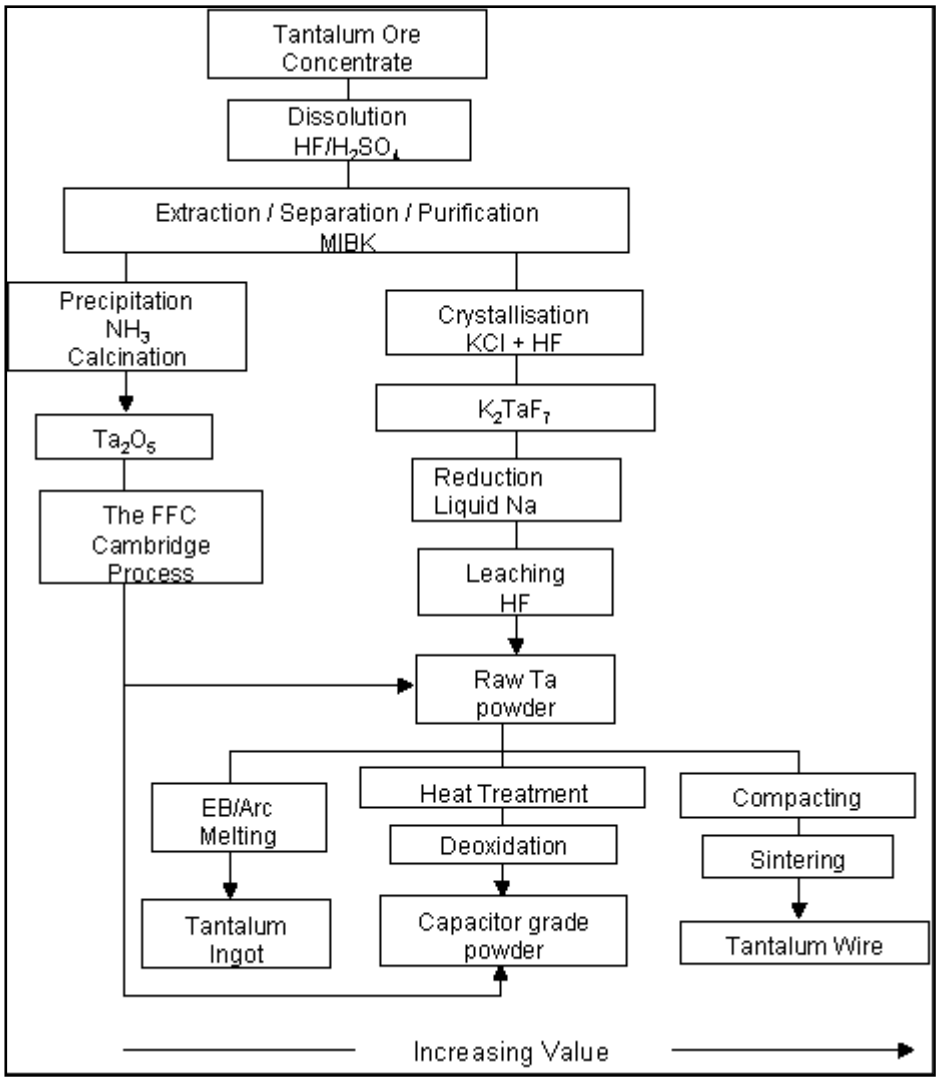
<u>Metalysis FFC reduction</u>

Anode:

$5O^{2-} + 2^{1/2}C \rightarrow 2^{1/2}CO_2 + 10^{-e}$

Cathode:
$Ta_2O_5 - 10^{e-} \rightarrow 2Ta + 5O^{2-}$

Figure 2:

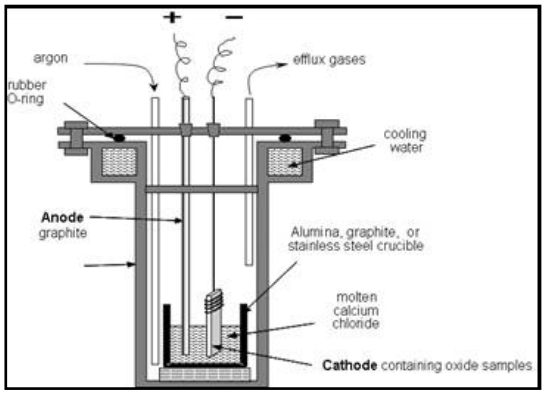


Metalysis FFC reduction cell

Figure 3 shows a design of the Metalysis FFC reduction cell. The basic Cambridge FFC reduction process is as follows:

- Calcium chloride is heated until the salt has turned in to a molten state.
- The electrodes containing the tantalum oxide pre-form, is lowered into the molten salt.
- Tantalum oxide pre-form should be fully immersed in the salt before reduction can take place.
- A set voltage is applied to the cell, typically below decomposition potential of the electrolyte, 1 to 3V.
- The inert gase, which in this case is argon, should be turned on before the reduction run takes place.
- After a set time the cathode is raised into the headspace and the cell is allowed to cool to room temperature.
- The salt around the reducted tantalum pellet forms a solid glass like barrier at room temperature, thus reducing oxygen pick up, on the tantalum particle surface.
- Last of all the cell is opened and the cathode is removed to the wash area, where the tantalum is separated from the cathode main body.

Figure 3:



Metalysis FFC powder morphology

During the FFC reduction process, from Ta_2O_5 to tantalum metal, the morphology of the tantalum metal is generated (figure 7). Figures 4, 5 & 6 also point to the fact that neck formation takes place within the reduction cell. This has a major advantage when you consider the other tantalum powder production routes. By growing the necks in a low oxygen environment, the necks are stronger then in a traditional production method, which is heat-treating the fine powder to form agglomerates. The heat treatment has the disadvantage of driving the oxygen in to the bulk and allowing the impurities to form at the grain boundary (neck site).
The Cambridge FFC reduction process can form different neck sizes depending on which CV/g capacitor grade powder Metalysis is aiming for.
It is considered that the structure grown inside the reduction cell will give the capacitor grade tantalum powder better electrical performance (DCL & surge) when compared to traditional tantalum production method.

Figure 4: Tantalum Metal

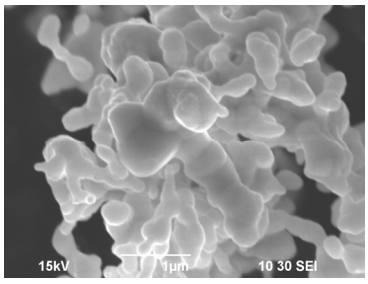


Figure 5: Tantalum Metal

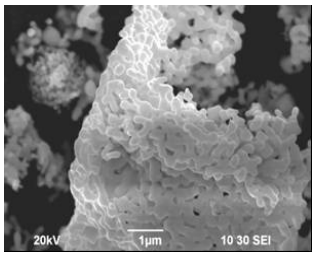


Figure 6: Tantalum Metal

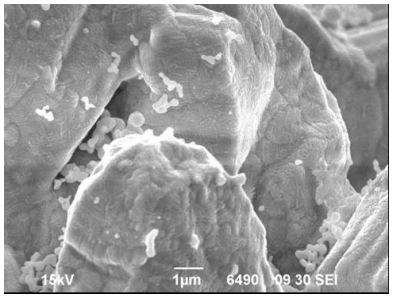
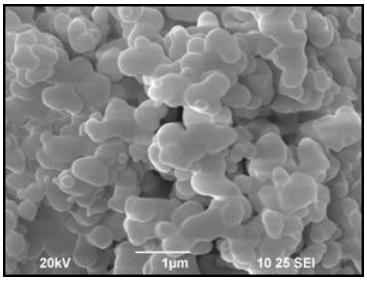


Figure 7: Tantalum Oxide



Physicals, Chemistry & Electrical Properties

Physicals

Advances in the physical properties are continually being achieved. The properties will also change due to the CV/g of the tantalum powder that Metalysis aim for. The examples are only a guide to the type of physical properties that Metalysis obtain on a regular bases with post processing.

- Scott density range of 1 to 2.5 g/ml.
- Crush strength of > 1Kg @ 5Dp.
- Flow range of 20 to 70 seconds (25g)

Chemistry

A number of elements are key to the quality Ta_2O_5 that can be grown at 2nm/V in a aqueous phosphoric acid (85 ^{o}C). The following trace elements levels can be achieved: C, Fe, H, Cr, Ni, Mg, & Nb are < 50 ppm.

Electrical

The electrical properties of capacitor grade powder will change with the CV/g specification aimed for.
A given example is shown below.

- Sinter Conditions: Vacuum $< 10^{-4}$mbar 1310 ^{0}C.
- Formation Conditions: 0.1% H_3PO_4, temperature 85 ^{0}C, Form at 30 volts.
- A value was obtained of 40kCV/g and a leakage of < 1nA/CV.

Conclusions

The Cambridge FFC reduction process is a unique process , which forms novel tantalum structures within the reduction cell. Because of this major advantage, the process has the potential offer the full range of CV/g capacitor grade powder (10k to 250K). The FFC process can also offer the industry a low environmental impact material, along with the ability of controlling the morphology of the tantalum powder.

Controlling the tantalum morphology will lead to a homogenous tantalum powder with good impregnation characteristics.

Acknowledgements

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References

1) Chen, G.Z.; Fray, D.J.; T.W. Nature 2000, 407,361
2) Fray, D.J.; Farthing, T.W, Chen, G.Z. PCT/GB99/017812, 1999.
3) Fray, D.J.J. of Metals 2001, October, 25-31.