



# NTNU

Innovation and Creativity

## **Titanium Production with the DeOx Process**

Ole S. Kjos

Department of Materials Technology

Supervisor:

Geir Martin Haarberg

# Outline

- Background
- Experimental
- Results
- Conclusion
- Thanks

# Background

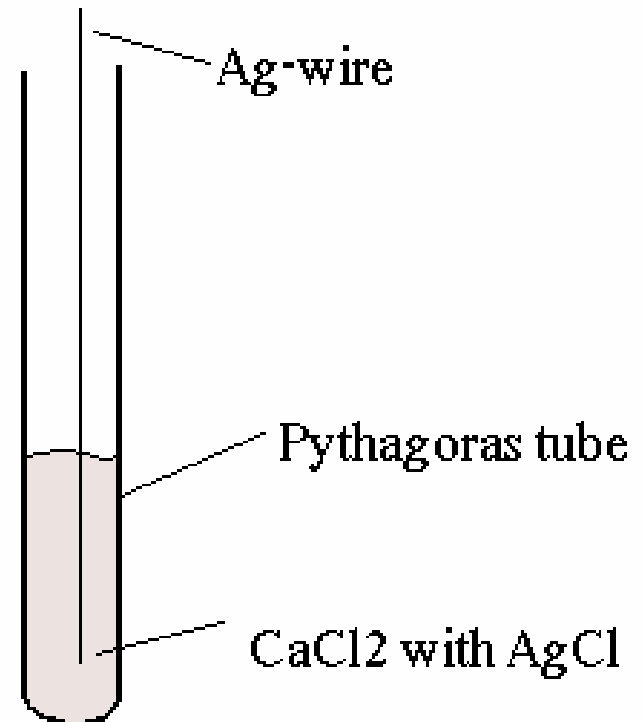
- There is a worldwide need for titanium
- Cheaper titanium would increase demand even more
- New, and more efficient ways of producing titanium are needed.

# Experimental

- All experiments were conducted in a closed laboratory furnace with argon atmosphere.
- The experiments were conducted with about 150g of  $\text{CaCl}_2$  based melt at 900 °C.
- 2.5 - 3g  $\text{TiO}_2$  was used as cathode preform.
- Carbon was used as anode.

# Reference electrode

- Ag/AgCl system was used as a reference.
- It was held in a mullite tube, sealed in the lower end.
- Reversible potential VS Ca-deposition from  $\text{CaCl}_2$  melt was calculated to be about 2.2 V.

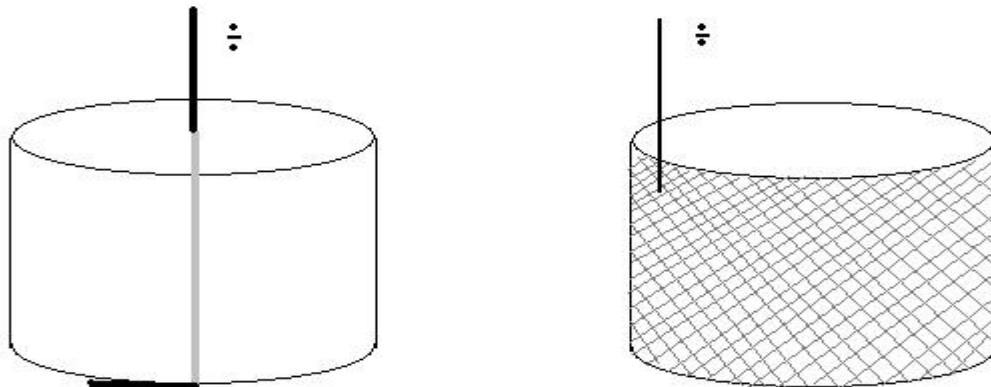


# The experiments

- First some experiments were done with constant cell-voltage at 3V.
- After the experimental setup was tested several experiment were conducted with a potentiostatic cathodic voltage of 1.4 V VS Ag/AgCl-reference.
- The same set of experiments was repeated with 1.8 V cathodic voltage.

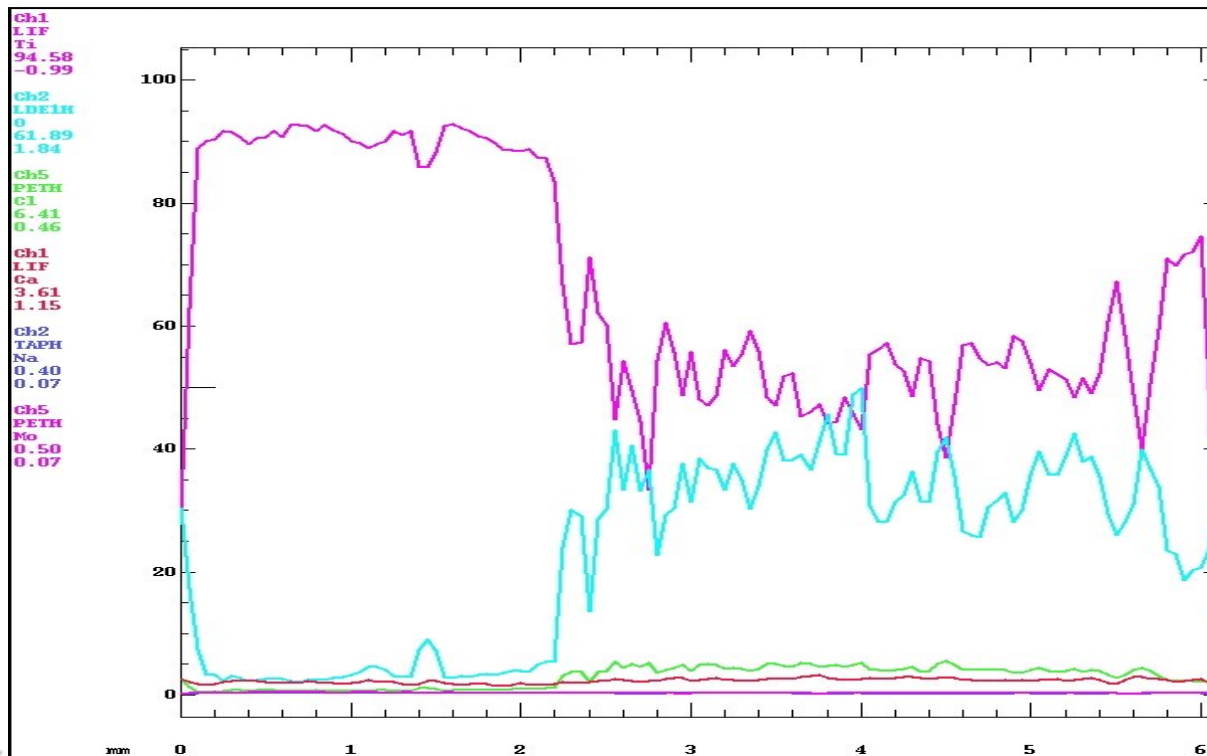
# The experiments

- For each cathodic voltage changes in the cathode assembly were made.
- After the experiments the final product was analysed by WDS or XRD.



# Results

- At 3 V cell voltage metallic titanium was produced. This is in agreement with literature.





# Results 1.4V

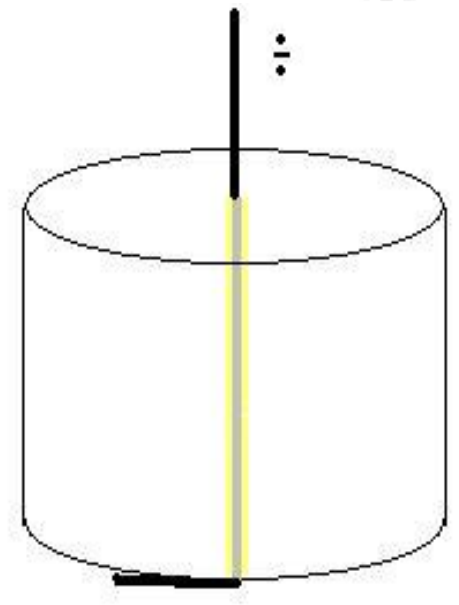
- At 1.4 V cathodic potential no metallic titanium was produced.
- At the start of the experiment the current was high (1-2A), but it rapidly decreased and stabilised at about 0.2A.
- The cathode was distinctly separated in two phases; calciumtitanat and titaniumoxide.
- No difference after 24 or 48 hours

# Results



# Results

- The calciumtitanate phase was always formed in the center of the pellet.
- The shell on the outer side of the pellet was always titanium oxide.
- The size ratio between them was similar when the size of pellet was changed.
- This did not change when the current was led through a mesh around the pellet.



# Results

- Probably spontaneous formation of calciumtitanate, and removal first happens around the edges.
- Results for 1.8 V cathodic voltage is practically identical to those from the 1.4 V series.
- Last run was 1.8 V for 24 hours, and then 2.0 V for 24 hours. This experiment gave metallic titanium, but with some oxygen.

# Conclusion

- It is possible to produce titanium metal with a DeOx-process.
- It was not possible to produce metallic titanium with 1.4 V or 1.8 V VS Ag/AgCl.
- Calciumtitanium-formation is probably spontaneous, but does not seem to be a problem.
- Changes in cathode size and metode for current connection done in these experiments did not result in significant changes in the product.

# Thanks to:

- My supervisor: Geir Martin Haarberg
- Kjell Røkke
- Christian Rosenkilde
- Sjur, SINTEF and the other people in the lab
- Elin Nilsen
- Ole Edvard Kongstein
- The other students working on the same project