High-Temperature Behavior of Tantalum Oxide Ta_2O_5

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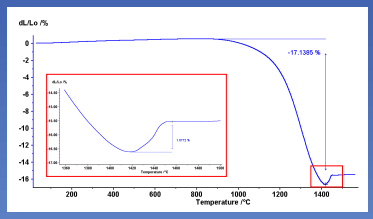

Fig. 1:
Sintering behavior of tantalum oxide
(orthorhombic β-phase)

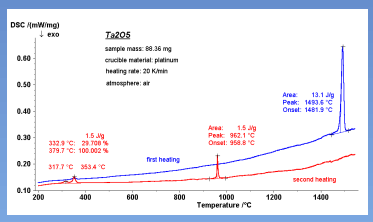

Fig. 3:
High temperature DSC experiment in air
(first and second heating under identical conditions)

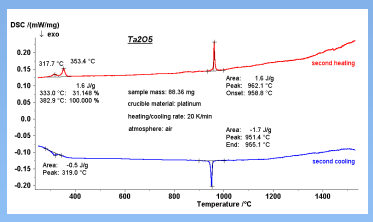

Fig. 5:
Reversible phase transitions of the Tantite
(second heating and second cooling)

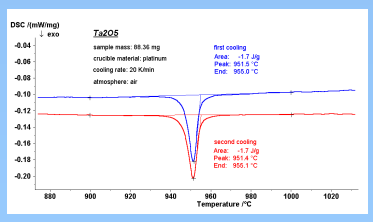

Fig. 7:
Reversible phase transition α to α' (tetragonal to monoclinic) during first and second cooling

The sintering behavior of tantalum oxide Ta_2O_5 (orthorhombic β-phase) was studied with pushrod dilatometry as depicted in fig. 1. The inserted zoom shows an unusual increase in sample length above the main sintering step.

A more detailed analysis of the high-temperature behavior of tantalum oxide was done by means of Differential Scanning Calorimetry (DSC). Further thermogravimetric experiments (TG) under the same conditions show no mass changes over the whole temperature range proving all DSC peaks to be phase transitions.

During the first heating in air up to 1550 °C at a heating rate of 20 K/min (fig. 3, blue curve) a peak was detected at 1482 °C (extrapolated onset). This endothermal effect with an enthalpy of 13.1 J/g is due to the phase transition from the orthorhombic β-phase (fig. 2) to the tetragonal α–phase. It is supposed to be the explanation for the effect found by means of the dilatometer experiment (fig. 1). This phase transition (β to α) is irreversible and occurred quantitatively which is indicated by the absence of an endothermal peak at about 1480 °C during second heating of the same sample under identical conditions (red curve in fig. 3). During cooling several phase transitions are detected yielding to the triclinic α''-phase of tantalum oxide (Tantite, fig. 4). The reversibility of these transitions is pointed out in figure 5 (second heat treatment: red curve, second cooling: blue curve).

The second heat treatment shows additional endothermal peaks at 350 °C and 960 °C which are the inversion to the observed peaks during the first cooling. The enthalpy of this reversible transition is only 1.6 J/g as depicted in fig. 7 (first cooling blue curve, second cooling red curve).

These phase transitions can be related to the sequence triclinic-monoclinic-tetragonal at 365 °C and 975 °C suggested by Waring et al. [3] for Ta_2O_5. The additional phase transition close to 310 °C (red curve in fig. 3) was not mentioned in the literature before.

Even though the structural changes related to the sequence α''-α'-α are very small, all phase transitions discussed above are also detectable with dilatometer experiments. Fig. 6 shows the thermal expansion of the tantalum oxide sample (α''-phase) during the second heat treatment (blue, first derivation: red). The coefficients of thermal expansion (CTE) are depicted for the α''-phase (100-288 °C) and for the α'-phase (360-930 °C).

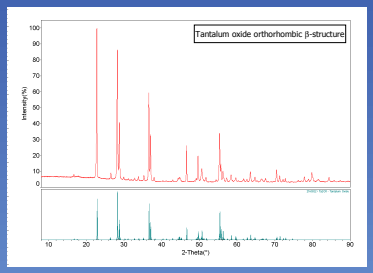

Fig. 2:
XRD-diagram of the orthorhombic low temperature structure of tantalum oxide (β-phase) [1]

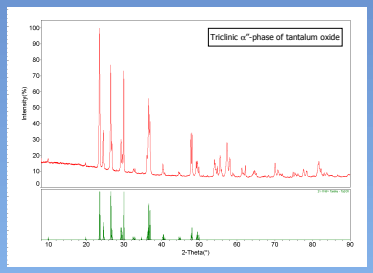

Fig. 4:
XRD-diagram of the Tantite
(triclinic α''-structure of tantalum oxide) [2]

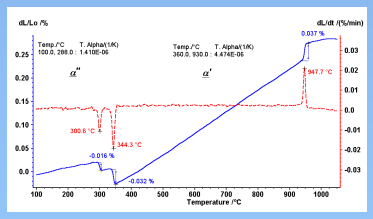

Fig. 6:
Results of the dilatometer measurement of Tantite
(second heating)

References:

[1] PDF#25-0922; J.Solid State Chem. 1970, 2, 445.

[2] PDF#21-1198; J.Res.Natl.Bur.Stand., Sect.A, 1968, 72, 175.

[3] J.L. Waring, R.S. Roth; J.Res.Natl.Bur.Stand., A, 1968, 72, 175-186.