

Effect of Sulphur and Phosphorus Microimpurities on The Features of Sodium-Reduced Tantalum Powders

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Tantalum metal is mostly used in the form of powders for capacitor making. One way of producing these powders is the reduction of tantalum by sodium from a melt containing K_2TaF_7 . The important condition of the powders application in capacitors is a low level of impurities that makes it necessary to use high-purity reagents for metal reduction. However, that reduces production of powders having insufficiently high specific surface. Therefore for increasing the specific surface of primary powders by change of particle growth conditions and stabilization of reduction conditions there is a usual practice to enter into the initial melt reagent additives containing boron, sulphur, phosphorus, nitrogen etc.

The aim of this study was to investigate the influence of sulphur and phosphorus microaddings contained in reagents employed or specially inserted into initial melt on characteristics of tantalum powders obtained by sodium reduction.

Powder reduction was carried out using laboratory installation in detail described elsewhere [1]. Our experiments have shown that sulphur addition into initial melt exerts noticeable influence on metal crystallization and size distribution of produced powder. As it is seen from the dependence represented in Fig. 1 the powder specific surface reaches 0.8-0.9 m^2/g at sulphur additions of 1.5-2.0 $\cdot 10^{-2}$ wt. %. An increase in specific surface of powder produced with sulphur addition has been stipulated by the rise in anode specific charge (Fig. 2, curves 1,3). As also seen from Fig. 2 sulphur is not an effective inhibitor in sintering process. For more complete use of high specific surface of the powder it is additionally doped by phosphorus in an amount of 0.005 wt. %. This allowed increasing the specific charge even more, especially at anode sintering low temperature.

The similar result was achieved by introduction of phosphorus microadding into the melt. In the Table 1 there are presented a bulk density (γ), a specific surface (S) and the particle average diameter ($\langle d \rangle$) calculated as the specific surface value of powders produced from melts with a molar ratio of $NaCl/K_2TaF_7$ equal to 6, the process temperature being 750°C. The Table 1 also represent anode specific charge of the powders sintered at 1550°C temperature. The data show essential influence of phosphorus present in the melt on the bulk density and the specific surface magnitude for tantalum powders.

Thus, the research done has shown that in the case of tantalum powders obtained by sodium reduction presence of sulphur or phosphorus microaddings in a melt renders essential influence to powder particles growth providing for powders with greater value of specific surface.

[1]. V.N Kolosov, V.M. Orlov, T.Yu. Prokhorova, A.T Belyaevsky. *Rasplavy*, No 2, p. 57-60 (2003).

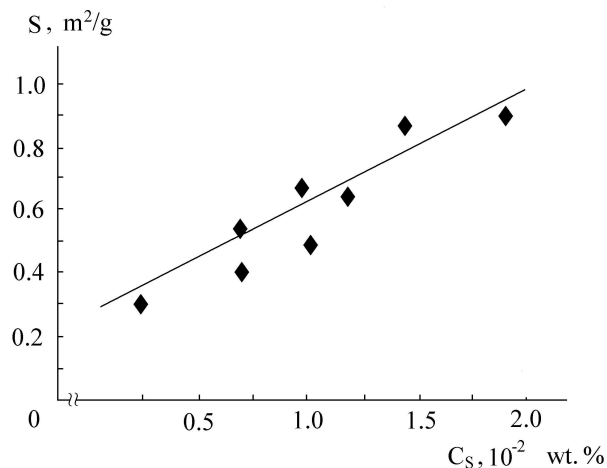


Fig. 1. Dependence of powder specific surface on sulphur content in an initial melt.

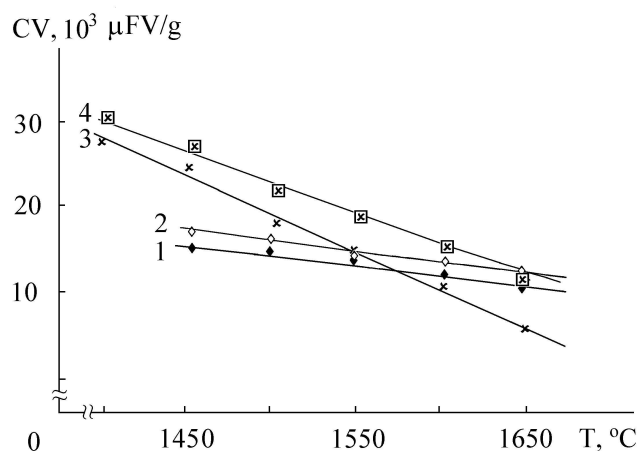


Fig. 2. Dependence of anode specific charge on sintering temperature: the powders obtained: 1 – without sulphur addition in a melt; 3 – ~ 1.5 $\cdot 10^{-2}$ wt. % of sulphur addition in a melt; 2, 4 – powders 1 & 3 additionally doped by phosphorus.

Table 1

Phosphorus addition influence on the characteristics of tantalum powders

Phosphorus addition	$\gamma, g/cm^3$	$S, m^2/g$	$\langle d \rangle, \mu m$	CV, $\mu FV/g$	CV*, $\mu FV/g$
Without addition of phosphorus	1,7	0,26	1,30	10830	13760
In K_2TaF_7 during its synthesis	1,4	0,40	0,90	14050	16380
In initial melt	1,1	0,98	0,35	19840	23530

CV* - powder additionally doped by phosphorus.