THEORY AND TECHNOLOGY OF SINTERING, HEAT TREATMENT, AND CHEMICOHERMAL TREATMENT PROCESSES

THE DEPENDENCE OF THE TECHNOLOGICAL PROPERTIES OF TANTALUM AND NIOBIUM ON THE PHYSICOCHEMICAL CHARACTERISTICS OF THEIR STARTING POWDERS

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New developments in electronics and other fields of engineering call for purer starting materials, in particular tantalum and niobium. In this connection, it has become necessary to improve further the technological processes of preparation of these metals. Among the most effective ways of achieving this is to subject tantalum and niobium to high-temperature vacuum refining with the aid of arc and electron beam melting.

The possibilities offered by the powder metallurgy of these metals have not been fully utilized either. By using suitable equipment and raising the vacuum in the high-temperature sintering of tantalum and niobium, it may be possible substantially to improve the quality of these metals in the dense condition. It should also be borne in mind that, in the majority of cases, inadequate purity of commercial niobium and tantalum powders prevents them from being used for the direct production of cast metal, without a vacuum sintering stage.

In this connection, vacuum melting conditions and the properties of the resultant ingots are determined to a large extent by the purity of the starting blanks (sintered bars) of tantalum and niobium. In turn, the purity of these bars and their technological properties (mainly ductility) are closely linked with the physicochemical characteristics of the starting powders. The establishment of such a relationship would be of considerable practical interest both as regards the production of various parts (strip, wire) from sintered tantalum and niobium bars and the vacuum remelting of these bars by some method, with the object of their further refining.

STARTING MATERIALS, THEIR PREPARATION, AND EXPERIMENTAL PROCEDURE

Electrolytic tantalum and niobium powders of different particle sizes and purities, initially mixed in accordance with a special calculation [1, 2], were generally used as starting materials in the investigations. In order to obtain the required particle size distribution, coarse electrolytic tantalum and niobium powders were hydrogenated, ground, and then dehydrogenated in a vacuum at 900°C. In some cases, use was made of sodium-thermic tantalum powders, as well as niobium and tantalum powders obtained from dense-metal scrap.

Bars 5 x 5 x 120 mm in size were compacted in a hydraulic press with vertical pressure, in a collapsible steel die. The compacting pressure was 43 kN/cm² for niobium and 63 kN/cm² for tantalum.

The compacted bars were subjected to high-temperature sintering in a laboratory vacuum retort-type apparatus with heating by the direct passage of electric current through the specimen [3]. The residual pressure after successive temperature rises did not exceed 0.133 N/m². The optimum sintering conditions were established by us earlier [1, 4]. Bars sintered by this procedure were forged into plates until the appearance of first cracks, after which the reduction ratio was measured on the bar height. The forged plates were rolled into strip, in which the amounts of the principal impurities were then determined. Some of the sintered bars were machined into standard specimens, which were subsequently subjected to tensile tests in a laboratory press with the aid of a reverser.

DISCUSSION OF RESULTS

Experience of the preparation of dense tantalum and niobium by the methods of powder metallurgy shows that the quality of sintered bars is governed chiefly by such physicochemical characteristics of the starting powders as the
The present investigation was undertaken with the object of determining the effect of these characteristics on the purity and ductility of sintered niobium and tantalum bars.

As was to be expected, the ductility properties (forging reduction ratio to the appearance of cracks) of sintered niobium bars depend on the particle size of the starting powder (Fig. 1). It is interesting to note, however, that the influence of particle size on the ductility properties of sintered bars decreases with decreasing purity of the starting powders. It can be seen from Fig. 1 (curve 1) that, as the mean particle size of niobium powder decreases by a factor of three (from 75 to 28 μ), the forging reduction ratio of sintered bars is nearly doubled. For tantalum (Fig. 2), this dependence is even more pronounced, which is presumably due to the higher melting point of this metal.

The optimum mean particle sizes of niobium and tantalum powders, obtained by the method of intersection of tangents to different parts of these curves, are very similar, and amount to 32 and 33 μ, respectively. As can be seen from Fig. 2, it is desirable that the maximum particle size of tantalum powder should not exceed 70 μ (i.e., that the whole powder should pass through the 200-mesh sieve), which is in agreement with data obtained by Driggs and Lilliendahl [5].

The use of coarser tantalum and niobium powders results in decreased ductility of sintered bars. This may be attributed to the lower density of bars sintered from coarse powders, since both grain growth and densification are inhibited by large pores. The pores remaining in such a case after sintering reduce the "live" cross section of the bar and intergranular contact, as a result of which cracks appear during forging at lower reduction ratios. The results of tensile tests carried out on sintered niobium specimens, which are shown in Fig. 3, are in full agreement with the conclusions arrived at in the technological tests. As in the above cases, with decreasing mean powder particle size, the strength of the bar markedly increases (Fig. 3, curve 1). As in the case of the ductility increase, the strength is approximately doubled as the mean particle size is reduced by two-thirds.

It might have been thought that the increase of strength was due to a higher impurity content of bars made from fine powders, but the chemical composition of all bars was practically identical.

It must be admitted that such results of the vacuum refining of niobium and tantalum could only be obtained by using a special procedure for calculating the optimum powder charge composition and sintering conditions selected...