A METHOD FOR PREPARING THE SUPERCONDUCTING COMPOUND Nb_3Sn

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A method for preparing the compound Nb_3Sn is described. The critical temperature of the specimens is 17.85° K and the width of the transition is approximately 1×10^{-2}° K.

THE preparation of a superconducting compound with a critical temperature of 18° K and transition width 0.03° K was described in a previous communication.[1] The method described below enabled a significant reduction in the width of the transition to be achieved.

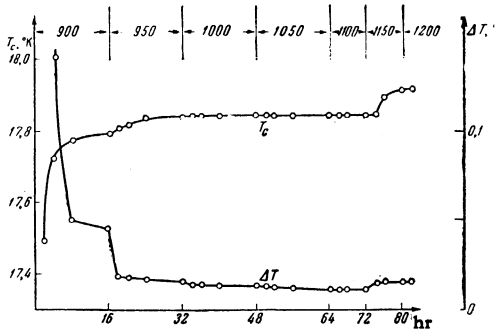
The compound Nb_3Sn was prepared from a powder containing 98—99% niobium with particle diameter 5—10 μ and a tin powder of the same grain size composition. Ethylene glycol was added to the powder mixture and the whole ground into a paste, from which specimens of different shape were prepared. After fashioning the specimens, the ethylene glycol was extracted by heating the specimen to 100° C (in a vacuum when convenient).

The heat treatment of the specimens essential for forming the compound Nb_3Sn was carried out in a gas envelope (a neon-helium mixture). Circulation of the gas at a rate of ~ 20 liter/hour was achieved by a membrane pump. The gas was cleaned of impurities just before entering the annealing zone of the furnace by adsorption on silica gel, cooled by liquid nitrogen. The gas envelope passed along quartz tubes inside the furnace. Contamination of the entering gas by diffusion and from the incandescent quartz was prevented by using concentric tubes, so that the returning gas passed along the intermediate space between the outer and inner tubes. A stainless steel tube with outer diameter 100 mm, inner diameter 20 mm and length 250 mm was placed in the furnace to achieve a satisfactory constancy and uniformity of temperature. The concentric quartz tubes with the preparation were placed in the mouth. In this way variations of temperature at different points of the specimen was reduced to a maximum of 5° K.

The reaction between the initial substances starts at a temperature somewhat above 800° C. However, heat treatment at higher temperatures is required to attain the best quality. Losses of tin by volatilizing are unavoidable on heating directly to temperatures above 1000° C. Thermal treatment therefore started at 850 or 900° C, with subsequent stepwise increase of temperature over a period of several hours.

In order to establish the results of the heat treatment, the transition point (the midpoint of the transition curve) and the width of the transition (the temperature difference between the points of intersection of the tangent at the point of inflexion on the transition curve with the straight lines determined by the fully superconducting and fully normal states) were checked, the magnetic susceptibility of the specimen being measured at 25 kc and for a magnetic field of 5×10^{-2} Oe after 2, 4, 8 and 16 hours of sintering anneal, after which the next temperature was attained and the process was repeated in the same order.

The results obtained for one specimen (a cylinder 3 mm in diameter, 50 mm high with annealing started at 900° C) are shown in the figure. The critical temperature T_c first increases. A steplike transition is observed at the first measurement of the annealing temperature (the temperature of annealing is shown at the top of the figure). The critical temperature reaches an almost constant value of about 17.85° K. A further increase in critical temperature is observed for annealing temperatures above 1150° C, possibly as a result of loss of tin.[2] The width of the transition region ΔT decreases with increase in the duration



of annealing. After sixteen hours of annealing at constant temperature the transition width practically changes no more. After the first and second change of annealing temperature a step-like transition to a narrower width is observed, but this process becomes less and less marked on increasing the temperature further. The width increases again for temperatures above 1150° C, evidently connected with the increase in critical temperature which takes place at the same time. The smallest transition width is 1.1×10^{-2}° K. For a preparation of the corresponding type with initial annealing temperature around 850° C, $\Delta T = 1.3 \times 10^{-2}$° K.

The x-ray density of the compound Nb_3Sn is 8.92 g/cm^3,[3] while the density of the specimens prepared by the method described is not greater than 3.5 g/cm^3, pointing to the appreciable porosity of the preparations, the pore volume reaching 60%. The apparent specific resistance of these preparations is 5×10^{-4} Ω cm at room temperature and is still 20% of this value at 20.4° K.

The critical field H_c, extrapolated on the basis of the shift in the critical point with transverse magnetic field, is about 165,000 Oe.[1] Due to the porosity of the preparations the value of the critical field determined from the critical current is considerably lower than this. The appreciable porosity of the preparations also leads to some phenomena which do not conform to the usual behavior of homogeneous superconductors.[4,5]

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