ANALYSIS OF X-RAY TRACES OF HIGH T_c Nb_3Ge AND Nb_3Si

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Résumé.- L'analyse des traces de rayons X de quelques films de Nb_3Ge obtenus par pulvérisation cathodique (début de transition à $T_c = 23,5$ K) présente une structure qui indique un accroissement de T_c lorsque la morphologie de croissance quitte les faces (100). Une anticorrélation de T_c avec la quantité de phase hexagonale est reportée. On donne aussi la valeur de a_0 pour quelques films de Nb_3Si .

Abstract.- Analyses of X-ray traces of some sputtered Nb_3Ge films (maximum T_c onset 23.5 K) show texture which indicates an improvement of T_c as the growth morphology moves away from (100) faces. An anticorrelation of T_c and the amount of hexagonal phase is reported. a_0 of some Nb_3Si films is also reported.

INTRODUCTION.- In this paper we present observations on two extensive sets of superconducting samples of Nb_3Ge and Nb_3Si with relatively high T_c 's made with a getter sputtering apparatus /1,2/. The first part is an analysis of X-ray data of a selection of Nb_3Ge samples with particular emphasis on the texture found in the films. A link is made between this texture and the presence of the hexagonal phase Nb_5Ge_3 which we have recently identified /2/ although it had been noticed (as the 'A' and '?' X-ray line) by previous workers /3,4/. The last part is devoted to lattice parameter measurements of some high T_c Nb_3Si .

EXPERIMENTS AND RESULTS : Nb_3Ge .- We present results of three main series of experiments (i), (ii), (iii); (i) refers to samples made with a composite target which have been discussed previously from a point of view of oxygen incorporation /2/, (ii) were made using an argon-arc melted target with Nb wire additions and ~ 50 ppm O_2 added to the argon sputtering gas, and (iii) were made with the same target and impurity levels as (ii) but with a modification to the target-substrate geometry and a slightly higher pressure than used in (i) and (ii) of about 600 mtorr. The data we present refers to runs in which the T_c onsets were above 19 K and for which a comprehensive set of X-ray traces were obtained. Of these samples 22 have had T_c onsets (1 % DC resistance) in excess of 22.6 K, 5 in excess of 23.0 K and one with an onset of 23.5 K. This indicates a degree of reproducibility but not a full specification of the optimum preparation conditions.

X-ray measurements, made on a vertical Phillips diffractometer, show that the relative in-

tensities of the X-ray lines is variable, and suggests that there is some texture in the films. However, it is very difficult from a single X-ray trace to obtain detailed knowledge of any texture in a film. We have concentrated on the intensities of the (200) and (211) lines, $I(200)$ and $I(211)$. Qualitatively we have noted that samples from (i) tended to have $I(200) \approx I(211)$; in an ideal A15 Nb_3Ge sample we may expect $I(211)/I(200)$ to equal 2.0 ± 0.2 /6/. Whereas $I(200)$ in series (iii) has been very much less than $I(211)$ ($< 1/10$). We found that the ratio of $I(211)/I(200)$ varies from sample to sample in a given run and hence in order to obtain a first approximation of a correlation we have chosen to plot the log of the average of $I(211)/I(200)$ versus the midpoint of the optimum sample of each run; we have taken T_c midpoints as these represent the average superconducting behaviour. The correlation in figure 1 is not very precise but, is an indication

Fig. 1 : Correlation of optimum T_c midpoints versus $\log$ (average $I(211)/I(200)$) for a given run

of a trend in all these samples. We comment that the correlation is one of general character of the texture being produced in the experiment as whole (in

some way). In series (iii) in which the orientation has moved from (100) toward (211) and (111) up to 30 % of the hexagonal phase has been observed. However it is clear that the T_c of these samples is anti-correlated with the amount of hexagonal phase produced as shown in figure 2. Our assessment of these

Fig. 2 : Plot of T_c midpoints of Nb_3Ge versus fraction of hexagonal phase present in films

results is that in order to obtain high T_c Nb_3Ge the orientation of the film growth must be away from (100) as preferred in (i) but as it approaches (111) a competing growth of hexagonal phase interferes with the formation of high T_c Nb_3Ge .

Nb_3Si .— Previously we have reported T_c onsets of 14 K in Nb_3Si /1/. We have now made several samples with onsets in excess of 16 K and one with an onset of 17.6 K using experimental conditions similar to series (iii) above, but otherwise as in /1/. For the best sample the X-ray trace shows 70 % of the A15 phase. a_o measurements of these "high" T_c films are shown in figure 3 plotted against T_c and a reasonable correlation is seen between decreasing a_o and

Fig. 3 : Plot of lattice parameter a_o of Nb_3Si versus T_c onset

increasing T_c . However we note that a_o is large, 5.185 Å for the best sample. This is much greater than the 5.08 Å /7/ expected from the Geller radii /8/. These expanded lattice parameters suggest that either off-stoichiometric samples can be made with varying degrees of defects, or that in our samples

a "high" T_c phase, undetected in our X-ray traces, is responsible for the superconductivity.

DISCUSSION.— Though the X-ray intensity measurements are open to some criticism it is easy to see possible mechanisms to explain the results. On a (100) growth face we would expect to see chains of tightly packed Nb atoms. On such a surface the Nb atoms are likely to attempt to pack with a spacing comparable to that observed in pure Nb, because such a surface monolayer would "carry" only part of the inter-atomic forces associated with the A15 phase. Hence one may expect such a growth morphology to give rise to defects in the chains. As one moves away from a (100) growth surface the numbers of exposed tightly packed Nb atoms are reduced and we speculate that it would be easier for the Nb atoms to arrive at their A15 sites. However if growth occurs on (111) then the hexagonal phase Nb_5Ge_3 seems to be nucleated and hinders the formation of the A15 phase. As yet we do not fully understand the way the main features of the sputtering conditions, especially in the series (iii) experiments, favours the growth near (211). We hope to report more fully on this in a forthcoming paper /5/. We comment that the same processes may be occurring in the Nb_3Si system as regards sputtering conditions and associated growth mechanisms.

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