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SUPERCONDUCTIVITY OF SINGLE-CRYSTAL $NbGe_2$*

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Summary

Small single crystals of $NbGe_2$ were grown by the hydrogen reduction of potassium-containing oxides. Measurements of electrical resistivity and superconductivity are given.

Matthias *et al.* [1] were the first to show that the A15 compound Nb_3Ge was a high transition temperature superconductor. Subsequent to the work of Gavaler [2] and later Testardi *et al.* [3] which pushed the T_c of Nb_3Ge to 23 K, Ghosh and Douglas [4] reported superconductivity to 16 K in sputtered films containing Ge and $NbGe_2$. It appeared that the Ge-rich portion of the Nb–Ge phase diagram might also be interesting from the standpoint of superconductivity, especially since Hardy and Hulm [5] found the C40 compound $NbGe_2$ to be normal to 1.2 K. The sputtering target used in ref. 4 had T_c = 1.85 K.

The work described here is based on the observation that $KNbO_3$ could be reduced to Nb metal in H_2 at relatively low temperatures. This suggested the possible production at relatively low temperature of Nb_3Ge by hydrogen reduction of $KNbO_3$ and K_2GeO_3 mixtures with a Nb:Ge ratio of 3:1. These mixtures were made both by ball milling the crushed oxides and by reaction of K_2CO_3 with a Nb_2O_5–GeO_2 mixture.

Hydrogen reduction was carried out overnight at temperatures in the range 800 - 1100 °C with little variation in the result. The mass that resulted from this reduction was interspersed with small columnar metallic crystals (typically 0.5 mm × 0.05 mm × 0.05 mm). A measurement of superconductivity on the entire product gave an incomplete transition with T_c onset of 2.0 K.

X-ray analysis of these crystals showed them to be $NbGe_2$ with lattice parameters a_0 = 4.965 Å, c_0 = 6.783 Å. A quantity of these crystals was isolated, crushed and found to have a T_c onset of 2.1 K and width 0.4 K. It

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*Dedicated to Professor B. T. Matthias in celebration of his 60th birthday.

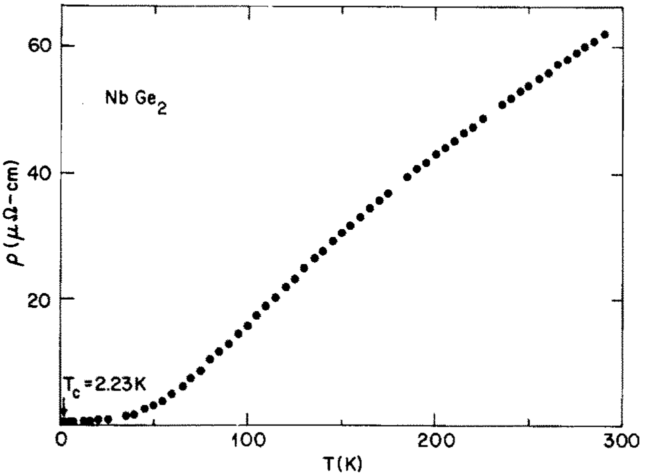

Fig. 1. Temperature-dependent electrical resistivity of an $NbGe_2$ single crystal. A similar crystal was found to have growth axis ⟨11$\bar{2}$0⟩, and this is quite probably the direction measured here.

is likely, therefore, that the T_c seen in the bulk reduction mass is due to $NbGe_2$. A four-probe electrical resistivity measurement on one of these crystals (Fig. 1) gave T_c = 2.23 ± 0.03 K. The resistance ratio to 4.2 K for this crystal is 100, the room temperature resistivity is 60 ± 15 μΩ cm. An X-ray rotation photograph of one of the crystals showed that its growth axis was ⟨11$\bar{2}$0⟩.

This result was compared with arc-melted samples at nominal compositions $NbGe_2$ and $NbGe_5$, annealed at 950 °C for one week. The $NbGe_2$ sample had T_c = 2.09 ± 0.02 K and for the $NbGe_5$ sample T_c = 2.16 ± 0.01 K. This last sample reacted somewhat with its protective Ta wrapper, possibly influencing the T_c. Nothing in the a.c. susceptibility was observed at higher temperature in either of these samples.

By varying the ratio of Nb:Ge in the initial mixture of oxides we were unable to produce material with a higher transition temperature. The actual mechanism for the growth of the $NbGe_2$ crystals is not clear. The possibilities include vapor growth and growth from KOH flux. It would be interesting to investigate this last possibility, but the container problem for molten KOH under these conditions is severe.

It is interesting that the hydrogen reduction of these oxides proceeds so readily, apparently due to the presence of potassium. This reduction technique might prove useful in the low temperature synthesis of a number of other intermetallics.

Acknowledgments

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