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A Simple Method for the Preparation of Wüstite Single Crystals

In recent years the electrical and magnetic properties of the transition metal oxides have received considerable attention. Much of the work has been aimed at the elucidation of the fundamental processes connected with the diffusion and the chemical reduction of these oxides. The use of single crystals in such experiments tends to simplify the analysis. The ready preparation of acceptable single crystals of the oxides has, however, proved to be a problem. The present letter describes a method for the preparation of wüstite single crystals which can also be used for certain other oxides.

Cech and Alessandrini [1] have described a technique for the preparation of single crystal films of wüstite. In this method evaporated $FeBr_2$ is oxidised to FeO in an atmosphere of H_2-H_2O. The oxide is allowed to condense on an MgO cleavage surface, where it is found to grow epitaxially when the substrate temperature is above 700°C. Since magnesium ions diffuse into the wüstite layer at this temperature it is difficult to remove a clean oxide layer from the substrate. The method is then, of limited use. Crystal growth from the melt is not advisable since crucible materials including platinum are heavily attacked by FeO melts. Crucible-free techniques for pulling oxide single crystals [2, 3, 4] are expensive and not always available.

During the course of an investigation into the early stages of the reduction of wüstite to iron [5], a very simple method for preparation of wüstite single crystals was developed. A strip of high purity iron was oxidised in a steep temperature gradient. The oxidising gas atmosphere contained 50% CO_2 and 50% CO. The lower part of the vertically suspended specimen was at 1450°C and the upper part at about 1250°C. The higher temperature exceeds the liquidus temperature of wüstite [6]. As a result of this, liquid oxide is formed which then drops into the lower part of the furnace tube. The specimen melts back until the temperature at its tip reaches the solidus temperature of wüstite. Grain growth takes place at the tip when the specimen is maintained at this temperature.

Fig. 1 shows large wüstite grains, prepared by this technique. The maximum grain diameter was found to be 20 mm after an annealing time of eight days. Plane surfaces formed at the tip of the oxide specimens as shown in fig. 2. Laue

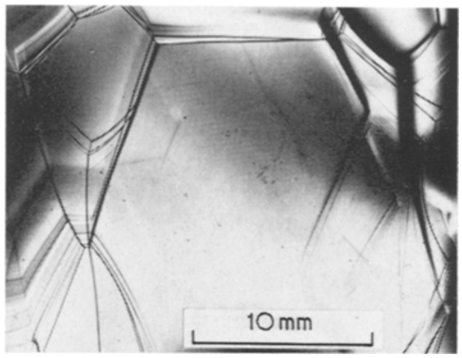

Figure 1 Polycrystalline wüstite (1390°C; 50% CO_2; 50% CO, 6 days). The large grain has a (230) surface orientation.

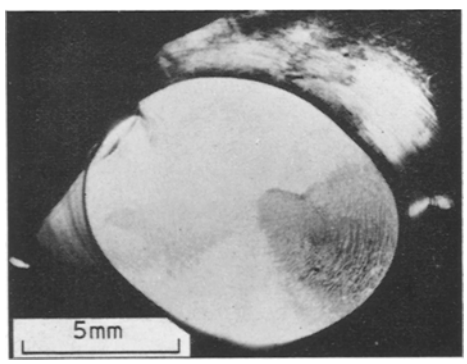

Figure 2 A surface with a (111) orientation at the tip of a wüstite specimen.

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back scattering photographs showed a (111) surface orientation formed in all cases.

The method described can also be applied to prepare large crystals of MnO, NiO and CoO. Since the melting points of the metals are lower than those of the corresponding oxides, the metal samples should be completely oxidised before the high temperature anneal.

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