A mild steel crucible as shown in Fig. 1 was used. It was made in two pieces to facilitate removal of the crystals. The entire sample was immersed in mineral oil during the growing process, but it was found unnecessary to keep the entire setup in a controlled atmosphere. By heating the top of the crucible and cooling the bottom, an initial temperature gradient of about 100 centigrade degrees over the 5 inch length was set up from bottom to top of the melted sample of sodium and the freezing took place from the bottom of the melt as the power to the heating coils at the top of the crucible was reduced. The large contraction of the sodium on freezing and cooling to room temperature made the removal of the grown boule very easy. There was no tendency of the sodium to wet the steel through the protective layer of mineral oil. The yield was four randomly oriented single crystals obtained in six growing attempts. For easy handling of the grown boules, a screw eye was threaded into the top of each. This end of the crystal was later discarded.

The chemical activity of sodium made it necessary to use rather extreme tactics to etch the boule to look for grain boundaries. At room temperature, the methanol etch followed by a rapid quench in xylene, which was successful in the case of lithium,[5] could not be applied to sodium because the action was far too rapid. We finally used as an etchant a mixture of roughly half and half commercial diethyl ether and methanol cooled to nearly liquid nitrogen temperature. Increased methanol content in the etchant gave faster action, less gave better etch pits. The procedure followed was to lower the test tube containing the etchant part way into a Dewar of liquid nitrogen, then to lower the boule into the mix. This etching process gave brilliant crystallographic blaze planes and a very bright metallic luster to the surface of the sample, on which grain boundaries were clearly visible.

Since all these observations had to be made while the sample was immersed and at low temperature, orientation of the crystals using the blaze plane optical reflections was not convenient. Once the boule was observed to consist of a single crystal, it was oriented using transmission Laue x-ray photographs of the crystal with the aid of the set of transmission Laue photographs of a body-centered cubic lattice published by Majima.[6] In addition, the high intensity of thermal diffuse scattering (TDS) simplified the analysis of the transmission Laue photographs of sodium. The blackening of the photographic film due to TDS traced out a geometrical figure with the same symmetry as that of the crystal relative to the x-ray beam.

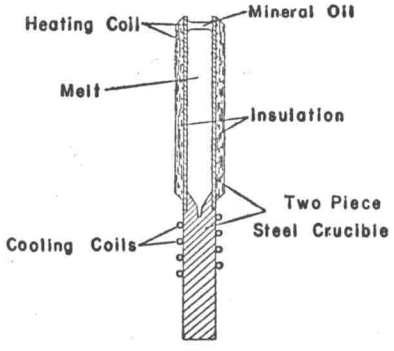

FIG. 1. Crucible for growing sodium crystals.

To prepare a specimen for the x-ray photography, a shim about a millimeter thick was cut off the boule using a string saw charged with a methanol-water mixture. It was possible to cut through the 0.75-inch diameter of the boule in about five minutes. While somewhat faster cutting can be done by charging the saw with pure water, it has been found that the addition of about 10 percent of methanol resulted in formation of a coating on the cut surface of the sodium which proved to be remarkably resistant to further corrosion by the air. With a little practice, it was possible to make cuts whose surface was flat to about 0.005 inch. The thinness of the x-ray specimen resulted in transmission Laue spots which were quite sharp and easy to locate.

Once the orientation of the crystallographic axes of the specimen was known relative to the boule axes, an acoustic specimen could be cut from the boule with any desired orientation. The advantage of growing large diameter single crystal boules is that one can cut reasonably sized oriented acoustic specimens from a single crystal boule of any orientation. The acoustic specimens were all about 2 cm in diameter and ranged in length from 0.9 to 3 cm.

We have found that sodium remains relatively unattacked if kept in contact with pure paraffinics, e.g., isopentane, mineral oil, Vaseline and paraffin, which have been treated by exposure to freshly cut sodium shavings. For example, holding the sample in the string saw mount is done by embedding the bulk of the crystal in Vaseline which has proven stiff enough to keep it in place for the cutting operation.

After cutting the crystal to proper orientation and approximate final size, it was cemented into an aluminum lapping ring[3] with pure paraffin. The flattening and polishing of the acoustic faces of the specimen were carried out using as a lap a piece of fine cotton sheeting moistened with methanol and stretched over a piece of plate glass. A final polish was imparted to the surface using mineral oil soaked cotton sheeting on plate glass. These techniques yielded acoustic faces parallel to about 0.001 cm in 1.6 cm. Samples which have been prepared, then stored long enough for a thin layer of oxide to appear on the acoustic faces can be cleaned up by stroking the crystal very lightly over mineral oil soaked cotton sheeting.

For the measurements to be made at high pressures, the quality of the mechanical bond between the quartz

[5] H. C. Nash and C. S. Smith, J. Phys. Chem. Solids 9, 113 (1959).

[6] M. Majima, Sci. Papers Inst. Phys. Chem. Research (Tokyo) 7, 259 (1927).