used in the 1-in. Westinghouse superconducting solenoid. Measurements of the absolute change of phase of dH–vA oscillations with pressure were made by the null method used in the later measurements on the noble metals. In the case of the alkali metals, of course, there were no problems of spurious phase shift due to intermodulation effects from other extremal cross sections of F.s. The measurements of dH–vA frequency were made with the automatic data recording system and computer processing techniques used by Chollet and Templeton[14] in studying the effect of alloying on the F.s. of copper.

2.3. Specimen Preparation and Handling

Specimen preparation, orientation, and handling were the most delicate parts of the experiment, for which we had to devise or adapt techniques to reach the goal of mounting a clean, undamaged, reasonably well-oriented and oxide-free cylindrical sample in the pressure bomb and of cooling this assembly successfully to liquid-helium temperatures. The difficulties naturally increased markedly as we progressed from potassium to cesium, but, in general, techniques learned for one metal could fairly easily be modified or adapted to suit the next, more active material. The initial procedures for potassium could be carried out under oil in an ordinary atmosphere, but for rubidium and cesium it was found preferable to do most of the handling under oil in a glove box filled with clean, dry helium. We used mineral oil that had been dried with sodium wire and then vacuum-distilled. A stock of this oil, into which fresh potassium wire had been extruded, was kept in a closed flask in the glove box. The glove-box atmosphere, particularly at critical times prior to the exposure of a dry crystal surface, was monitored by cutting slices from a block of potassium and observing the rate of deterioration of the exposed surface. The removal of any traces of moisture diffusing into the box was found to be at least as important as removing oxygen, and a refrigerating coil was used at critical stages to do this. Under the best conditions a cut surface of potassium would remain bright for 15–30 min, while under oil in an open dish the surface would remain bright for a week or more.

The single crystals were required finally to be some 4 mm long and 2 mm in diameter. A selection of glass tubes, each a few inches long and drawn down to have very thin ($\sim$0.1 mm) walls and an internal diameter of rather over 2 mm, were used as simple moulds to provide the required cylindrical shape. A small phial of high-purity metal was cracked open and placed in a beaker of oil, which was then warmed on a hot plate to just above the appropriate melting temperature. By means of a rubber bulb, several suitably sized beads of molten metal could be drawn (after some practice!) into a sample tube, which was then laid in a shallow dish of cold oil. The inclusion of several beads of metal in each tube provided additional protection to the inner samples against atmospheric corrosion during the x-ray orientation process, which had to be done outside the inert atmosphere of the glove-box. After several tubes of samples had been prepared, the shallow dish of oil in which they lay was warmed gently to some 10°C above the melting point of the metal and was then covered, laid on an insulating pad, and allowed to cool

very slowly. The solidified beads of metal usually showed striations that were apparently related to crystal orientation, so the crystalline uniformity of the various beads could, to some extent, be judged by eye.

Since the experimental samples were to be mounted in a fixed holder with its axis along the magnetic field, only the axial orientation of the crystal, as defined by its cylindrical shape, was of importance. The x-ray determination of this orientation was carried out with the samples still in their original tubes, the ends having been plugged with dry petroleum jelly injected by a hypodermic syringe and finally capped (outside the glove box) with Apiezon Q-compound and a coating of Glyptal cement. The orientation measurement is somewhat complicated in the alkali metals by their rather large interatomic spacing, combined with the very high absorption by both glass and metal of those rather low-energy x rays that can be diffracted through a sufficient angle to give a useful Laue diffraction pattern. However, by using rather thin glass and allowing the x-ray beam to strike only the very edge of the sample, we were able to make reasonably satisfactory transmission Laue pictures.

In preparation for an experiment a tube containing a suitable crystal was returned to the glove box. The coil and pressure bomb had already been introduced via a small hole in the wall of the box and temporarily sealed in place with a split stopper around the beryllium–copper pressure capillary. The glass tube was then carefully cracked and the chosen crystal allowed to slide out into a dish of oil. Then, handled with spatulas of thin card, the specimen was washed in dry xylene and etched gently in xylene containing a little amyl alcohol until, when tried for size under oil, it would just slide into a 2-mm test hole. The etched specimen was finally washed in xylene, allowed to dry briefly, and slid into the pickup coil. The crystal was allowed to rest in position on the base plug of the coil, since it was felt that the light spring normally used would damage the soft metal. The pressure bomb was then screwed into place and the assembly removed from the glove box. The pressure seal was then finally tightened, the external field coils were connected, and the bomb, with a small overpressure of helium gas on the specimen, placed in the cryostat and allowed to cool fairly rapidly to liquid-air temperature. The experimental procedures from that point have already been described.

3. RESULTS AND DISCUSSION

Four specimens of potassium were successfully mounted and run. All gave good signals, but one showed a long beat in the dH–vA oscillations that indicated that this sample was a bicrystal. Rubidium gave good signals from one and fair signals from another of three specimens, while cesium gave good signals in two cases out of seven. Details of the specimens and of the experimental results are given in Tables I and II. In Table III we list our value for the zero temperature bulk modulus B_0 (the reciprocal of the volume compressibility) and the values of atomic volume V_0 corresponding to our values of F_0, the dH–vA frequency for a free-electron sphere. We compare these with the values given by conventional