SOME OBSERVATIONS OF SURFACE PHENOMENA APPEARING ON LEAD SINGLE CRYSTALS

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In the course of preparation of some single crystals of lead according to the method of Chalmers (2), the occurrence of {111} and {100} crystallographic surfaces under certain conditions of freezing was observed. Single crystals of lead were grown in a horizontal graphite boat in air, with the oxide film being removed occasionally by scraping the liquid surface with a preheated glass rod. By using care to introduce a minimum of thermal and mechanical disturbance, a crystal with very little oxide film could be obtained. The lead used was stated to be of 99.999+% purity. Two growth procedures were employed: in the first, the boat was suspended so as to reduce mechanical vibration of the melt to a minimum; in the second, constant vibration was applied to the boat so as to freeze a solid having a rippled surface.

In the first type of experiment a characteristic step-like structure forms on the top surface of the crystal if a {111} plane lies slightly out of coincidence with the mean top surface of the melt. The upper surface of the solid at the interface tends to grow forward parallel to a {111} plane during advance of the interface until further extension is limited by sudden formation of a step in the surface. The extending {111} plane tends either to rise above or to penetrate below the free liquid surface, depending on its angle with the horizontal. The height of the liquid meniscus is appreciably greater than the height of the steps which are formed. This is illustrated schematically in Figs. 1(*a*) and 1(*b*).

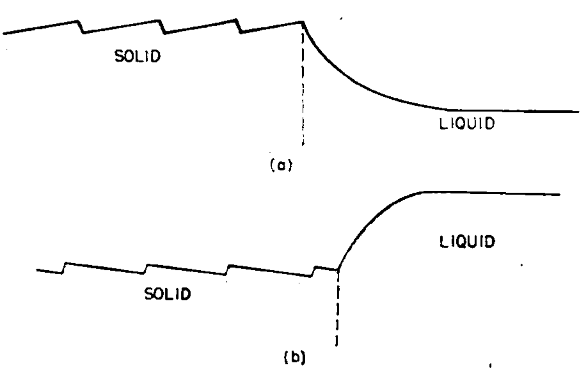

Fig. 1. Advancing interface with surface steps forming (not to scale).

In the second type of experiment, a constant vibration of low amplitude was applied to the melt. As a consequence of this, a dense pattern of ripples approximately 0.2 mm. wide and transverse to the boat axis was frozen into the crystal surface. The surface so prepared was found to have highly directional reflectivity for visible light, with reflection maxima appreciably sharper than those from etch pits in the same metal. The poles of the planes producing the reflections were located by means of an optical goniometer of the type

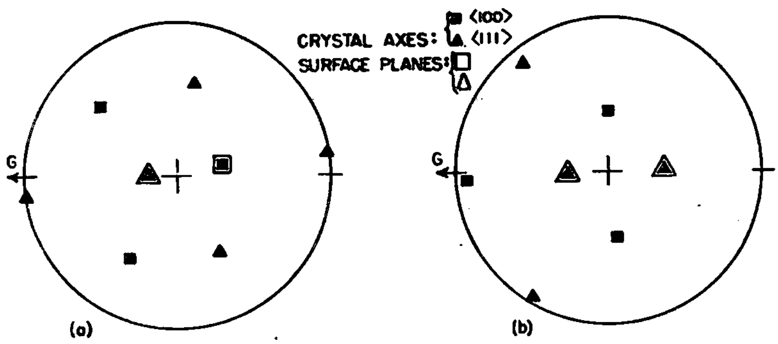

FIG. 2. Stereographic projections of two crystals, showing crystal axes obtained by X-ray diffraction (solid points) and axes of surface planes producing optical reflections (open points). Arrow at *G* shows growth direction.

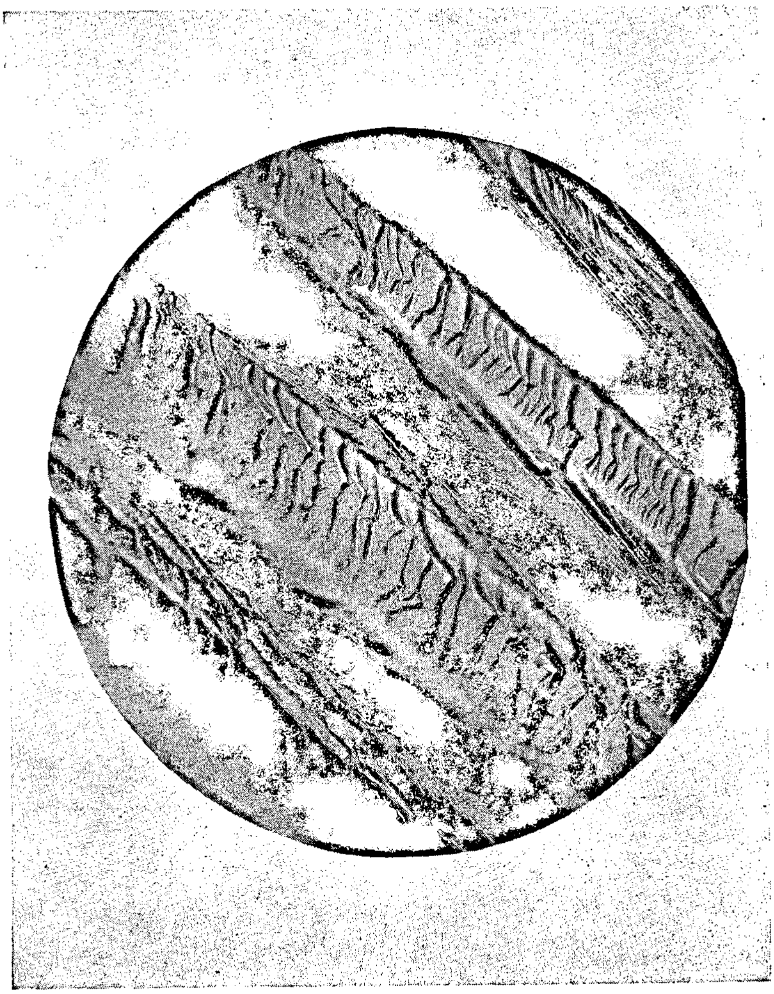

FIG. 3. Micrograph of rippled surface (85×).

described by Barrett (1). The poles thus obtained are shown superposed on the stereographic projection of the corresponding crystal region in Figs. 2(*a*) and 2(*b*), for two different crystal surfaces. (The axis of the containing boat is horizontal in these diagrams, and growth direction was right to left.)

In each case, optical reflection maxima were found corresponding to reflections from those {111} planes of the crystal which lay most nearly parallel to the local top surface. In case 1(*a*), one of the {100} planes was also thus favorably oriented, and produced a strong reflection as well. The angular resolution of the reflections was good enough for striations (4) occurring in case 1(*a*) to be readily separated in angle.

Under magnification, the frozen ripple surfaces appear to be composed of segments of plane facets. Fig. 3 shows an example of strong faceting on the crystal of 2(*a*) (magnification 85×). The conspicuous plane segments in this figure correspond to {111} planes on the side of the ripple toward the melt. The plane facets on the side of the ripple away from the melt are less extensive in area, both for the {100} planes in case 2(*a*) and for the {111} planes in case 2(*b*).

The occurrence of characteristic surface structures arising in the freezing of a metal has also been reported by Elbaum and Chalmers (3), who found that the interface exposed by decanting the liquid from freezing lead single crystals exhibited a step-like structure when a {111} or {100} crystal plane was within approximately 15° of the mean plane of the interface.

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