

FIG 2.—Top, X-ray diffraction pattern of dental amalgam before application of pressure. Diffraction peaks corresponding to  $\gamma_1(Ag_2Hg_3)$  and  $\gamma_2(HgSn_{7-8})$  are labeled. Bottom, X-ray diffraction pattern of the same amalgam immediately after the application of 50 kb pressure shows an increase in intensities of  $\gamma_2$  peaks, which corresponds to reorientation or recrystallization of  $\gamma_2$  crystals. Surfaces of sample were removed by grinding; thus, X-ray diffraction patterns correspond to internal amalgam structure.

face of the sample. Since the surface of dental amalgam usually has a higher mercury content from the action of condensation, unreacted alloy is seldom seen there. Also, since this alloy does not contain zinc, the phase disappearance is not catalyzed by the presence of small amounts of zinc.

Further investigation of this phase disappearance was performed using optical and scanning electron microscopy. Samples were polished slowly under cold water down to 0.05 µm alumina and were etched according to Wing's<sup>7</sup> procedure. Photographs were taken with a Jeolco scanning electron microscope. Figure 5 (top left and right; bottom

*left*) shows the grain structure of one sample; Figure 5, *bottom right*, shows the structure of the matrix of another sample after pressure had been applied.

Three distinct crystal types can be seen in Figure 5, top left (orig mag  $\times 2,100$ , on a 4  $\times$  5 inch negative): (1) an unreacted alloy particle,  $\gamma(Ag_3Sn)$ ; (2) a  $\gamma_2(HgSn_{7-8})$  crystal growing in the grain boundary between two  $\gamma$  grains; (3)  $\gamma_1(Ag_2Hg_3)$  crystals that belong to the matrix. The identification of these phases was made by holding the electron beam fixed in the middle of each crystal and using nondispersive techniques to obtain the energy spectrum of the scattered

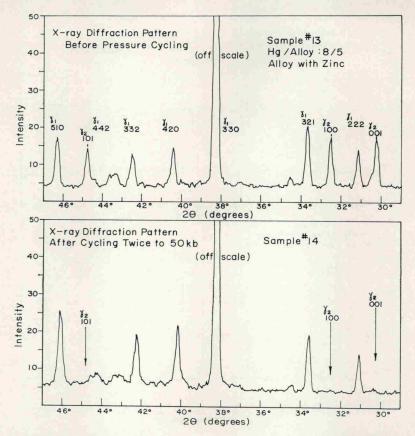


FIG 3.—Top, X-ray diffraction pattern of amalgam before application of pressure. Sample contained mercury-to-alloy ratio of 8:5, alloy contained zinc. Bottom, X-ray diffraction pattern of identical sample shows  $\gamma_2$  peaks have disappeared six months after cycling twice to 50 kb. There is no evidence of formation of new phase.

X rays. This identification is considered tentative until additional microprobe analyses can be performed.

Figure 5, top right, shows the region that contains  $\gamma(Ag_3Sn)$  and  $\gamma_2(HgSn_{7-8})$  at orig mag  $\times 8,000$ ; the crystal boundaries, shapes, and textures can be clearly seen. No similar  $\gamma_2$  crystals were found in the samples that had been subjected to high pressure. Figure 5, bottom left, presents the grain structure of  $\gamma_1(Ag_2Hg_3)$  in the matrix between unreacted alloy particles. Comparison with Figure 5, bottom right, shows that the application of high pressures fractures, severely cold works and plastically deforms the crystals in the sample.

## Discussion

Although a pressure-induced phase transformation could occur in  $\gamma_2$ , new diffraction peaks would be detected in the X-ray traces if the transformation were irreversible. Since the  $\gamma_2$  phase disappears in some of these samples after several months to a year, this phase disappearance is probably a diffusion dependent phenomenon rather than a pressure induced phase transformation. Solid state reactions are often accelerated by cold working and plastic deformation. The activation energy for atomic diffusion may be lowered in plastically deformed regions, yielding an increase in the diffusion rate. Also, the process of deformation may produce a large temporary increase in the num-