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## Reversible single-crystal rocking-curve broadening.\* By L. C. CHANG, T. A. READ and M. S. WECHSLER, Columbia University, New York, N.Y., U.S.A.

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Previous reports (Chang & Read, 1951; Chang, 1951, 1952) have been made on the characteristics of the Au-Cd alloy system with particular emphasis on transformation behavior. The work described here was performed in connection with an investigation of the non-equilibrium behavior of Au-Cd after heat treatment. It was found that the rocking-curve width increased reversibly and isothermally as a result of heat treatment during which the specimen remained in the  $\beta$  phase (CsCl structure).

A 50 atomic % single-crystal specimen of Au-Cd ( $2 \times 0.5 \times 0.08$  cm.) was quenched to room temperature after having been annealed for 30 min. at 480° C. With a Geiger-counter spectrometer, the variation of rocking curve width of the 100 and 200 reflections was then observed at 35° C. as a function of time after quench. Cu K $\alpha$  radiation monochromated by second-order reflection from the cleavage plane of calcite was used and the widths were calculated by taking the ratio of the integrated intensity to the peak height of the rocking curve. As shown in Fig. 1, the widths of the two reflections



Fig. 1. Rocking curve width v. time after quench, Au-Cd, 50 atomic %, annealing temperature 480° C., quench temperature 19° C., temperature at which measurements were made 35° C.

increased in the same manner but the percentage spread in sin  $\theta$  did not. This is interpreted as indicating that the

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broadening of the reflections with time after quench is due to a spread in the orientation of mosaic blocks rather than a spread in the value of lattice parameter. The receiving slit of the Geiger counter was sufficiently wide so that, for all positions of the specimen as it was rotated through the Bragg angle, all the reflected radiation for a given reflection was received by the Geiger counter. Under these conditions if the broadening were due to a variation of the lattice parameter, we would expect  $\Delta \sin \theta / \sin \theta$  to be nearly equal for the two reflections since  $\Delta \sin \theta / \sin \theta$  would depend chiefly on  $\Delta d/d$  and not on the order of the reflection or the Bragg angle. Since it was observed that the values of  $\Delta \theta$  for the 100 and 200 reflections were much more nearly equal than the values of  $\Delta \sin \theta / \sin \theta$ , it is concluded that the cause of the broadening is primarily a spread in the orientation or size of mosaic blocks.

The reversible nature of the effect is indicated by the fact that if the specimen is reheated and quenched again, the rocking-curve widths are initially narrow and increase in the same manner as described above. Similar measurements were made on the same specimen after a slow cool from the annealing temperature. It was found in this case that the rocking-curve widths were relatively narrow, characteristic of the specimen in the as-quenched condition, but no changes occurred upon isothermal hold. Observations of the same type of broadening were also made at  $26^{\circ}$  and  $57^{\circ}$  C. The data reveal that the rate of rocking-curve width increase with rising temperature of isothermal holding is approximately  $2\frac{1}{2}$  times for each  $10^{\circ}$  C. rise in temperature.

It seems likely that the change in rocking-curve width with time is related to the relaxation of the quenching stresses which remain after the rapid cooling. It is suggested that plastic deformation during cooling is confined to the warmer and hence softer interior of the specimen, so that the mosaic block disorientation in the surface layers immediately after quenching is no greater than that which is present after slow cooling. The inhomogeneous deformation leads to residual stresses, however, and these may subsequently cause some plastic deformation in the surface layers and the subsequent increase in the disorientation of mosaic blocks.

## References

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