TDPAC Study of Quenched Single Crystal of Cadmium

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Lattice defects such as vacancies or interstitials in Cd or Zn have been extensively studied by TDPAC method.\textsuperscript{1-4}) Usually, irradiation by energetic particles or cold working is employed to introduce lattice defects into the specimen. Since both vacancy type and interstitial type defects are introduced by these methods, a clear cut identification of the specy of a defect which becomes mobile in a particular range of temperature is difficult with employing these methods of defects introduction. For the case of the quenching experiments, however, it has been known that only vacancy type defects are introduced into the specimen. Therefore the comparison of TDPAC spectrum in a quenched specimen with that in an irradiated or cold worked specimen is quite helpful to identify the specy of a defect in question.

Withuhn\textsuperscript{1}) et al. have applied the TDPAC measurements method for both irradiated and quenched Cd and have observed essentially the same spectrum around at the annealing temperature of 120 K. From the results they have a conclusion that vacancy type defects become mobile at around 120 K, namely, the stage III in Cd. In a later report by the same group\textsuperscript{2}), however, the vacancy model has been questioned based on their experimental observation that the components $f^1$ and $f^3$ in their TDPAC spectra, which are assigned as vacancy type defects in Ref. 1, are reduced in Ref. 2 if one minimizes the influence of cold working during the quench. In Ref. 2, they have suggested that $f^1$ and $f^3$ in Ref. 1 might have been caused by the cold working during the quench and not by quenched-in vacancies. In a recent report\textsuperscript{3}), however, they support the view in Ref. 1, vacancy type defect migration in stage III, based on a new experimental result.

A similar quenching experiment have been performed with applying single crystal of Cd in the present, of which results will be reported in the followings. The motivation of the work is to study the structure of the defect-probe atom complex by measuring the TDPAC spectra as a function of the crystal orientation as has been performed by Seeboeck et al.\textsuperscript{4}) or by the present author.\textsuperscript{5})

5-nine or 6-nine Cd single crystals are prepared by freezing them from one end in an Ar atmosphere. The size of the crystal was $20\times 5\times 1$ mm$^3$. The crystal was irradiated by a proton beam to introduce $^{111}$In by $^{112}$Cd(p,2n)$^{111}$In reaction at 77 K. The specimen was annealed at RT for about 10 days to anneal out radiation damage produced by the irradiation. It has been known that the annealing treatment is sufficient to anneal out all defects due to the irradiation. Using the thick target approximation for the radioactivity
production and assuming that the beam is uniform in the irradiated area, the concentration of $^{111}$In atoms is estimated as $3 \times 10^{-10}$ at the end of the irradiation, which decays with half life of 2.81 days of $^{111}$In. This concentration of $^{111}$In is much lower than that of the vacancy in equilibrium at the melting point of a metal, typically $10^{-5}$-$10^{-4}$. Therefore it is expected that almost all $^{111}$In can be trapped to vacancy type defects even when only part of the equilibrium vacancy are quenched into the specimen.

The specimen was kept at 588 K (6 K below the melting point of Cd) for 5 min. and dropped into liquid nitrogen. The specimen was set in a specimen holder in liquid nitrogen without any warming up after the quench and TDPAC spectrum was measured at 77 K. After the measurement of the as quenched state, the specimen was isochronally annealed at 120 K and 140 K for 10 min. and TDPAC spectra were measured at 77 K as a function of the crystal orientation. The whole procedure was repeated 3 times, one of the results is shown in Fig. 1. As can be seen from the result for the as quenched state, the spectrum is orientation dependent. This shows that the crystal is a single crystal. Also the spectrum is that of the $^{111}$In at the substitutional site of Cd without trapping any defect or impurity. Upon annealing the specimen at 120 K or 140 K, almost no noticeable change takes place in the spectrum. They show the same spectrum with that of the as quenched state if compared at the same orientation. The same results are obtained in the other two experiments. Namely, in the present quenching experiment, no positive evidence of the vacancy migration or trapping to $^{111}$In atoms is obtained between 77 K and 140 K. This is in contrast to the result by Witthuhn et al.\textsuperscript{1} One possible cause of the discrepancy may be in the difference in the thickness of the specimens used in the experiments. Witthuhn et al. have used 30 μm thick polycrystals for their quenching experiment, which is much thinner than those used in the present, (1 mm). The quench rate, therefore, is expected much higher in their experiment to freeze in the vacancy to 77 K but not so in the present.

Further work using a thin single crystal will be necessary to realize the purpose of the present experiment.

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References


5) Hanada R. in Ref. 3.

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**Fig. 1.** TDPAC spectra for quenched single crystal of Cd. The spectrum should be compared at the same angle $\alpha=90$ to examine the annealing effect. The crystal c-axis is oriented in an arbitrary direction relative to $K_1$. 

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