PAC Spectrometry of Quenched Cadmium. A Detailed Annealing Study

著者	Hanada R.
journal or publication title	CYRIC annual report
volume	1993
page range	37-41
year	1993
URL	http://hdl.handle.net/10097/49740

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Introduction

Among several methods of point defect introduction, technique of quenching, fast cooling of heated specimen into cold fluid, is the simplest one. Different from radiation damage or cold work method, only lattice vacancy is introduced by the quenching method. So the interpretation of the experimental result should be less complicated than the cases of damage or cold work, where both self-interstitial and vacancy are introduced at the same time. In a previous report by the present author$^{1)}$, where PAC spectrum using ^{111}In as a probe was measured, several new Fourier components have been observed during the isochronal annealing between 100 and 160 K, which has been ascribed to the defect-probe complex, namely multiple vacancies trapped to the probe atom ^{111}In.

In the present study, a similar annealing experiment has been performed, although a more detailed (2K/10 min.) annealing schedule was employed contrary to the previous measurement(4K/10 min). The purpose is to study the annealing behavior of the defect component rather continuously with the annealing temperature and to find one defect component may switch to another in some temperature range. This may demonstrate the nuclear method of PAC is powerful to detect a process that a defect-probe complex attracts another defect forming a new complex. Such a process has never been observed by conventional classical methods such as the resistivity measurement and yet possible for PAC, where the change in EFG (the electrical field gradient) upon the formation of new complex is sensitively reflected in the nuclear precession frequency.

Experimental

The methods of specimen preparation, quenching, annealing and PAC spectrum measurement are the same with those described previously.

Fourier analysis of PAC spectrum was performed for the first 2^8 points (300 nsec) with doubling and adding 2^9 null data to obtain 2^{10} points. Hamming window was applied to suppress the contribution from the spectrum at large delay time. The new feature of the present analysis is the addition of null data, which does not cause any change in the spectrum and yet increases the Fourier spectrum points leading more smooth Fourier spectrum[2)].

Result

Figure 1 shows PAC spectra for a pure Cd specimen quenched from 543 K (270 C) during the isochronal annealing between 77 K and 180 K. Figure 2 is the corresponding Fourier spectra. The presence of substitutional components (S1-S3) or the defect components (D1 and D2) in some temperature range is the same with those reported[1)]. The new feature of the present result with the finer annealing schedule is the gradual change of D1 component(170 MHz) into D1' component (155 MHz) between 126 K and 138 K. Since D1 has been interpreted as due to an ^{111}In atom trapping one vacancy[1,3)], it is very likely the new D1' component is as due to an ^{111}In trapping multiple vacancies (most likely two vacancies). Namely one more vacancy arrives at the ^{111}In-single vacancy pair thus forming ^{111}In-two vacancies pair between 126 and 138 K. The defect components observed in the present are summarized in Table 1 together with those by others and by the present author. The D7 component by the present author in a previous report turns out to be a variation of D1 as observed in the present experiment and hence renamed as D1' in Table I.

Discussion

With the present light of the experimental results by PAC, it is worth discussing the past results of Cd defect study by resistivity measurement.

A large resistivity decrease has been observed between 110 K and 140 K after electron[4)] or neutron irradiation damage[5)], which has been named as stage III. On the contrary after a quench, only a minor resistivity decrease (in some case the increase) has been observed in stage III region[6)] and a major resistivity recovery has been found between 140 K and 180 K[7)]. With the lack of the knowledge of species of defect causing resistivity change, it was inevitable for some authors to conclude that vacancy migrates between 140 K and 180 K and other type of defect migrates in stage III[8)].

The present PAC result reveals that the quenched-in vacancy migrates in stage III(110-140 K) thus forming ^{111}In-vacancy pair (D1) or ^{111}In-multi-vacancy complex (D1'). This result is in agreement with the result of the resistivity recovery after irradiation damage[4,5)]. Namely, vacancy migrates in stage III and annihilates with interstitial clusters, which have been formed at lower temperatures by the migration of self-interstitial.

Then why no resistivity recovery takes place in stage III after the quench ? This may be answered as follows. After the quench only vacancy is present without interstitial or interstitial clusters. The most likely process after the quench in stage III is, therefore, the formation of vacancy complexes(di-, tri-, multi-vacancies). Namely, vacancy migrates in stage III and meets with other vacancies and ceases to migrate. This process, however, causes only a minor resistivity change since the vacancy complexes still continue to contribute to the resistivity. The break-up of these complexes and annihilation to sinks take place above 140 K which is observed as the major resistivity recovery. This break up and annihilation of the vacancy conplex above 140 K is consistent with the present PAC result that the annealing of D2 component, which is ascribed to vacancy clastens formed around ^{111}In[1,3], also takes place in the same temperature range.

Although only vacancy type defect is introduced by the quenching technique, the annealing process is rather complex as shown by the present PAC study where many defects components are observed during the annealing. A simple-minded interpretation of the resistivity recovery result, where a major recovery stage is *assumed* as corresponding to vacancy migration and annihilation, sometime leads to a wrong conclusion.

At present the proposed interpretation of ^{111}In-vacancies complex (^{111}In-di-vacancy pair) for D1' is still a speculation and further study as measurement of the angular dependence of the component will be necessary to examine the structure.

Acknowledgement

The author would like to thank to the staff of the CYRIC for their cyclotron operation and the beam transport. He also thanks to prof.Gary S. Collins (Washington State University) for his suggestion improving our Fourier analysis during his visit in IMR.

References

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Table 1. Summary of defect components in PAC spectrum for quenched pure cadmium.

Component	ω_Q /Mrads^{-1}	Temp.Range/K	proposed species
S1[1,3,*]	125	77-140 180-RT	^{111}In at sub.site
S2	250	1st harmonic of S1	
S3	375	2nd harmonic of S1	
D1[1,3,*]	170	110-135	^{111}In-single V
D2[1,3,*]	10	130-160	^{111}In-V-clusters
D3[3]	0	130-160	^{111}In-Stacking fault
D4[1,*]	45	120-140	^{111}In-multi-V
D5[1]	195	-	
D6[1]	215	-	
D7(D1')[1,*]	155	130-140	^{111}In- a few V.

[1]:ref.[1] ,[3]:ref.[3], * : This work.

The D7 in ref.[1] is renamed as D1' in the present.
With the present 3-detectors, D3(the unperturbed term,S_0)can not be detected.

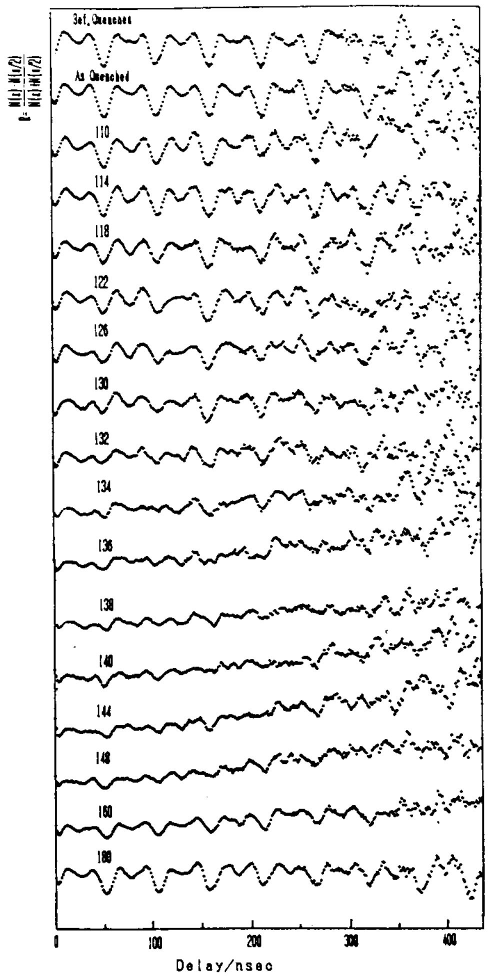

Fig. 1. PAC spectrum of ^{111}In in quenched pure Cd.
T_Q: 270C. 10 min.isochronal annealing. Measurements at 77 K.

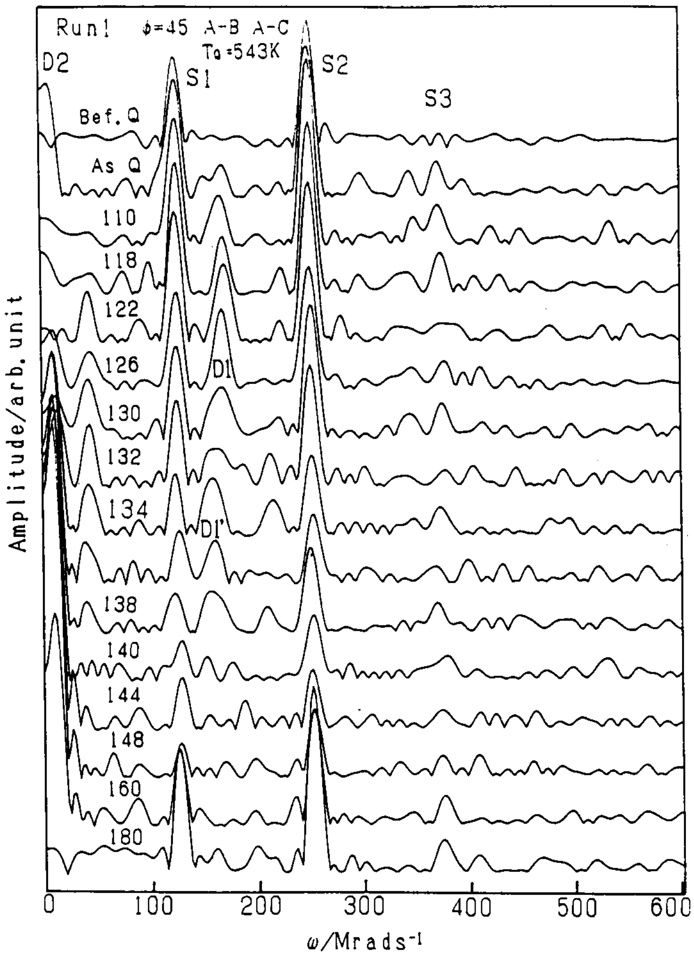

Fig. 2 Fourier spectrum of Fig. 1. Note that D1 shifts to D1' at the temperatures between 126-134K.