# Positron-annihilation studies of stable Al-based icosahedral quasicrystals

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The positron-annihilation lifetime spectra in the Al-Pd-Re alloy system, the positron-annihilation Doppler-broadening spectra by slow variable monoenergetic positron beam in $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$ alloys, and positron-annihilation lifetime spectra by slow positron beam in $Al_{71.5}Pd_{20.3}Mn_{8.2}$ were measured. These measurements indicate that every sample contains a dense distribution of structural vacancy-type sites. This result is consistent with our previous expectation of the relationship between the presence of vacant centers in clusters and the stability of icosahedral quasicrystals. Furthermore, the structural vacancy densities in icosahedral quasicrystals of $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$ have been estimated to be $7.7\times10^{20}$ and $5.0\times10^{20}\,cm^{-3}$, respectively. [S0163-1829(99)08109-6]

## INTRODUCTION

Recently, many proposals have been reported for an atomic cluster model of Al-based quasicrystals (QC's), e.g., icosahedral Al-Cu-Fe,[1] icosahedral Al-Li-Cu,[2] icosahedral Al-Pd-Mn,[3,4] etc., by single-crystal x-ray-diffraction analysis, single-crystal neutron-diffraction analysis, and high-resolution transmission electron microscopy and so on. However, a unified view of the structure has not yet been obtained because of insufficiency of quantitative experimental data with unsatisfactory *R* factors. The purpose of the present study is to obtain some structural information about structural vacancies, and to give complementary information to x-ray analysis in icosahedral QC's.

We have already indicated by positron-annihilation measurements that many kinds of stable icosahedral QC's contain a dense distribution of structural vacancy-type sites without exception since the early work by Kanazawa and co-workers.[5–11] For instance, in icosahedral Al-Li-Cu,[6] Al-Pd-Mn,[10,11] Al-Cu-Fe,[9] and Al-Cu-Ru (Ref. 9) QC's, it has been clarified that most positrons are trapped at intrinsic structural vacancies. Especially, in the case of plastically deformed Al-Pd-Mn (Ref. 10) alloys, no additional lifetime component could be detected, because the structural vacancy density is so high that the detection of an effect of dislocations on the positron lifetime which is usually observed in crystalline metals is hindered.

Kimura *et al.*[12] reported that 12 atoms icosahedra consisting of Al atoms have a covalent bonding nature; that is, the quasicrystalline structure of Al-based QC's is stabilized by coordinating the atom clusters with vacant centers quasiperiodically. Positron-annihilation measurement is the most powerful method for detecting such vacancy-type sites in the icosahedral QC alloys and the amorphous alloys because their detection is independent of the structural periodicity unlike the diffraction method. We have discussed experimentally the relationship between the existence of vacancy centers of atomic clusters in QC's and their thermal stability through the positron-annihilation experiments,[7] and our measurements are consistent with the picture by Kimura *et al.*[12] Actually, in all the stable samples, positron-annihilation measurements detect structural vacancies sensitively as mentioned above.[6,9–11] On the other hand, in the case of metastable icosahedral QC, the positron-annihilation measurements[5,8] suggested that centers of Mackay icosahedron in Al-Mn-Si and Al-Mn-Si-Ru QC's are occupied with atoms such as manganese.

In this study, we report positron-annihilation lifetime measurements for icosahedral Al-Pd-Re alloys with widely different electrical resistivities, Doppler-broadening measurements by slow variable monoenergetic positron beam for icosahedral $Al_{70.7}Pd_{21.34}Re_{7.96}$, $Al_{71.5}Pd_{20.3}Mn_{8.2}$, and $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$, and positron-annihilation lifetime measurement by slow positron beam for $Al_{71.5}Pd_{20.3}Mn_{8.2}$, and then discuss the relationship between the existence of vacant centers of atomic clusters in QC's and their thermal stability. Furthermore, by positron-annihilation Doppler-broadening measurements by slow positron beam, the structural vacancy density has been estimated for icosahedral $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$.

 

## EXPERIMENT

Al-Pd-Re alloy ingots with various compositions having various resistivities, selected from the phase diagram of Al-Pd-Re system in the region of the quasicrystalline phase found by Sawada *et al.*,[13] were prepared by arc melting the compressed raw material powder after uniformly mixed in an argon atmosphere. Then the ingots were annealed for 12 h at 1213 K in vacuum after sealing them in quartz tube. Subsequently, the quartz tubes enclosing the ingots were quenched in water. $Al_{71.5}Pd_{20.3}Mn_{8.2}$ and $Al_{68.31}Mn_{21.21}Si_{10.48}$ ($\alpha$-phase) alloy ingots were produced by arc melting in an argon atmosphere, and then annealed for 12 h at 1023 K and for 5 h at 773 K, respectively. The quality of the quasicrystalline phase and the $\alpha$ phase were confirmed by x-ray-diffraction analysis.

The positron-annihilation lifetime measurements in icosahedral Al-Pd-Re alloy system were carried out at room temperature. The positron source ($^{22}$Na activity about 5 $\mu$Sv), sealed in a thin foil of Kapton, was mounted in a specimen-source-specimen sandwich. The positron-annihilation lifetime spectra were recorded with a fast-fast coincidence system employing H2431Q photomultiplier by Hamamatsu and $1\times1$ in$^2$ $BaF_2$ scintillators. The time resolution of this system was 230 ps full width at half maximum (FWHM). For each spectrum at least $1.0\times10^6$ annihilations were counted. The time resolution function was assumed to be composed of two Gaussian functions. Using this time resolution function, the lifetime in the bulk of well-annealed pure Al (purity 99.9999 wt %) was measured as $165\pm2$ ps. Each $\chi^2/q$ was below 1.2. After subtracting the background positron-annihilation lifetime spectra were analyzed using POSITRONFIT.[14]

The positron-annihilation lifetime measurements by slow positron beam in icosahedral QC $Al_{71.5}Pd_{20.3}Mn_{8.2}$ were carried out at room temperature by use of the positron pulsing system with an intense slow positron beam generated by an electron linac in Electrotechnical Laboratory (ETL) LINAC facility. The lifetime spectra were obtained by measuring the time interval between the timing signal derived electrically from the pulsing system and the timing signal of an annihilation $\gamma$ ray detected with a $BaF_2$ scintillation detector. The detailed lifetime measurement system is described elsewhere.[15] For each spectrum, at least $3.0\times10^5$ counts were accumulated. The positron-annihilation lifetime spectra were analyzed using the ''RESOLUTION'' routine[16] with good variance of the fits with a time resolution of about 290 ps FWHM.

The slow variable monoenergetic positron beam for Doppler-broadening measurements is composed of a $^{22}$Na positron source (about 5 mCi) and a single W ⟨100⟩ foil of 1 $\mu$m thickness. The foil was annealed at 2273 K in a vacuum of $\sim10^{-9}$ Torr, and was attached in front of the source to moderate the positrons. A fraction of these energetic positrons emitted from a $^{22}$Na source is thermalized through a variety of collision processes in a tungsten moderator and are re-emitted from this moderator as monoenergetic slow positrons. The intensity of the slow positron beam is about $1.0\times10^4$/s. The incident positron energy is variable from 0 to 13 keV. The measurements of Doppler-broadening spectra by slow positron beam were carried out at the room temperature by use of a solid-state detector (pure Ge). The total counts in a spectrum corresponding to each incident positron energy were $8.0\times10^4$.

TABLE I. Positron lifetimes for the Al-Pd-Re alloy system. Experimental uncertainty is $\Delta\tau=\pm3$ ps.

| Alloy | $\tau_1$ (ps) | $\tau_2$ (ps) | $I_2$ (%) |
|---|---|---|---|
| $Al_{70.7}Pd_{21.34}Re_{7.96}$ | | 213 | All trap |
| $Al_{73}Pd_{18}Re_9$ | | 218 | All trap |
| $Al_{73}Pd_{19}Re_8$ | | 215 | All trap |
| $Al_{72}Pd_{20}Re_8$ | | 218 | All trap |
| $Al_{72}Pd_{21}Re_7$ | | 213 | All trap |

In order to estimate the vacancy-type defects in the specimens, the *S* parameter, which was determined by the ratio of the central area over 10 channels to the total area of the positron Doppler-broadening spectrum after subtracting the background, was used. Then, the experimental results were analyzed by applying the scaling method.[17–19]

## RESULTS AND DISCUSSION

Table I shows the lifetimes of the quasicrystalline phase $Al_{70.7}Pd_{21.34}Re_{7.96}$, $Al_{73}Pd_{18}Re_9$, $Al_{73}Pd_{19}Re_8$, $Al_{72}Pd_{20}Re_8$, and $Al_{72}Pd_{21}Re_7$ having orders-of-magnitude different resistivities. The positron-annihilation lifetime spectra of all the samples are composed of a single component; a fit with a two-component spectrum has shown that the intensity of the second component is below about 1% with respect to the first component. For all the samples an almost equal lifetime of $215\pm3$ ps is observed irrespective of the sample resistivity. The values of these lifetimes support that the annihilation site corresponds to the vacant site. Since the lifetime spectra are composed of a single component, it is thought that most thermalized positrons are trapped at vacant sites.

We have measured the change of the positron-annihilation lifetime in icosahedral QC $Al_{71.5}Pd_{20.3}Mn_{8.2}$ by slow positron beam of variable energy. The raw data for the positron-annihilation lifetime spectra at various positron incident energies are shown in Fig. 1. These observed lifetime spectra

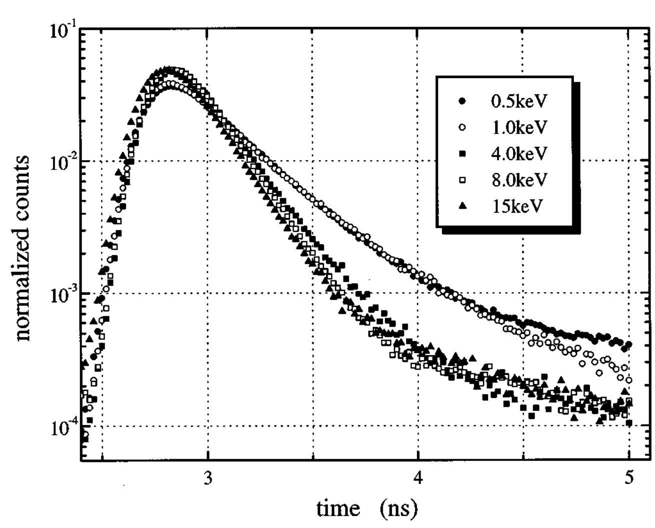


FIG. 1. Positron-annihilation lifetime spectra for $Al_{71.5}Pd_{20.3}Mn_{8.2}$ by slow positron beam.

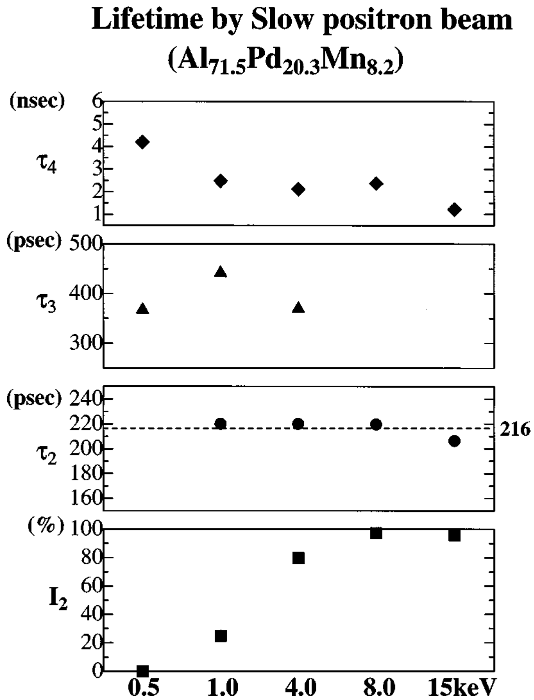


FIG. 2. Positron-annihilation lifetimes and intensity for $Al_{71.5}Pd_{20.3}Mn_{8.2}$ by slow positron beam.

are well fitted with two or three lifetime components. Figure 2 shows the values of the lifetime of component four $\tau_4$, lifetime of component three $\tau_3$, lifetime of component two $\tau_2$, and its intensity $I_2$ at positron incident energies 0.5, 1.0, 4.0, 8.0, and 15 keV. The observed lifetime $\tau_4$ at 0.5, 1.0, 4.0, 8.0, and 15 keV and lifetime $\tau_3$ at 0.5, 1.0, and 4.0 keV are in the range from 1 to 4 ns and in the range from 350 to 450 ps, respectively. The lifetimes longer than 600 ps have been generally accepted as resulting from annihilation of ortho-positronium[20] (o-Ps) trapped at a surface state, so the obtained lifetime $\tau_4$ is attributed to o-Ps and the value of lifetime $\tau_3$ corresponds to that of pickoff annihilation on the surface. The lifetime $\tau_2$ of main component at 1.0, 4.0, 8.0, and 15 keV is $213\pm7$ ps. It is considered that this component corresponds to that of vacancy-type defects. It should be noted that the intensity $I_2$ increases gradually and becomes ~97% at 8.0 and 15 keV. This means that most thermalized positrons are trapped at the vacant site in the course of diffusing to the surface at the positron incident energy of 8.0 and 15 keV; in other words, the structural vacancy density in icosahedral QC $Al_{71.5}Pd_{20.3}Mn_{8.2}$ is so high that the thermalized positrons cannot diffuse a long distance. As already mentioned in QC Al-Pd-Mn most thermalized positrons are trapped at vacant site.[10] Hence, the result by the slow positron beam is consistent with the results of the conventional positron-annihilation lifetime measurements. It is generally accepted that the lower limit for vacancy concentrations in metals to saturate the positron trapping is the order of $10^{-4}$.[21] From this point of view, it is thought that the magnitude of the structural vacancy density in icosahedral QC Al-Pd-Re and Al-Pd-Mn is larger than the order of $10^{-4}$.

In order to discuss the structural vacancy density in icosahedral QC Al-Pd-Re and Al-Pd-Mn in detail, we have measured the positron-annihilation Doppler-broadening spectra by slow positron beam in icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$, $Al_{71.5}Pd_{20.3}Mn_{8.2}$, and $\alpha$-phase $Al_{68.31}Mn_{21.21}Si_{10.48}$. Recent positron-annihilation lifetime measurements[8] have already clarified that the center of Mackay icosahedron in $\alpha$-Al-Mn-Si is empty. Considering the behavior of positron diffusion in $\alpha$-Al-Mn-Si, the most thermalized positrons are trapped at the vacant site when entering within the region of a certain radius larger than the size of vacancies, which we call the positron trapping radius. The positron trapping radius in $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$ has been determined by starting from the structure proposed by Elser and Henley.[22] According to Elser and Henley, the $\alpha$-Al-Mn-Si crystal structure is composed of a bcc packing of Mackay icosahedra with vacant centers, whose lattice constant is 12.68 Å and the structural vacancy density is calculated at $9.8\times10^{20}$ cm$^{-3}$. So, we have applied the scaling method[17–19] to the structure based on Elser-Henley model as known. In the process of diffusing from bulk to surface, the positron mean diffusion length in $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$ is represented as a function of the positron trapping radius by calculating the probability which the thermalized positron diffuses without trapped at vacant sites. As described previously, since the positron-annihilation lifetime spectrum of $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$ is composed of a single component with a lifetime corresponding to the vacant center of Mackay icosahedron, the calculated $S$ parameter for $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$ is written as $S=S_sF_s+S_dF_d(F_s+F_d=1)$, where $S_s$ and $S_d$ are the value of $S$ parameter for surface and defect, respectively. $F_s$ and $F_d$ are the annihilation rate for surface and defect, respectively. In this way, the positron trapping radius in $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$ is determined by fitting the calculated $S$-parameter data to the measured one.

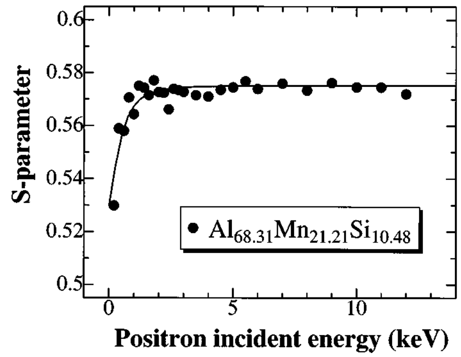


FIG. 3. The measured (closed circles) and calculated (solid line) $S$-parameter data for $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$.

Figure 3 shows the measured $S$-parameter data for $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$. It is seen that the values of the measured $S$-parameter increase rapidly from 0 to 2 keV and is saturated. The rapid increase from 0 to 2 keV reflects that the positron diffusion length in $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$ is very short. The solid line in Fig. 3 represents the fitted $S$ parameter, in which the positron trapping radius is taken to be 4 Å.

We assume that the most thermalized positrons are trapped at vacant-center sites of Mackay icosahedra in icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$ selectively with the same positron trapping radius as the one in $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$. Namely we assume that the trapping radius in icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$ is 4 Å. Though we know that the icosahe-

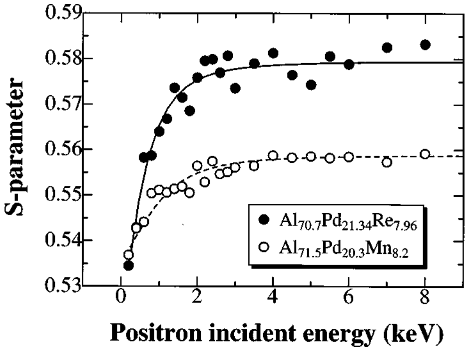


FIG. 4. The measured (closed and open circles) and calculated (solid and dashed lines) *S*-parameter data for icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$.

dral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{7.15}Pd_{20.3}Mn_{8.2}$ have the structure with Mackay icosahedron clusters being arranged quasiperiodically, the density of trapping sites for icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{7.15}Pd_{20.3}Mn_{8.2}$ are determined by using the trapping radius of 4 Å assuming to an approximation that the structure of icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$ is composed of bcc packing of Mackay icosahedron with vacant centers by fitting the calculated *S*-parameter data to the measured one. Figure 4 shows the measured *S*-parameter data for icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ (closed circles) and $Al_{71.5}Pd_{20.3}Mn_{8.2}$ (open circles). It is seen that the values of the measured *S* parameter for both samples show a similar tendency, signifying a dense distribution of vacancy-type sites. The solid and dashed lines in Fig. 4 represent the fitted *S* parameter for icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$, respectively. From the fitting to the measured *S* parameter, the lattice constants for icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$ are determined as 13.75 and 15.85 Å, respectively. The structural vacancy densities for icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$ and $Al_{71.5}Pd_{20.3}Mn_{8.2}$ are then calculated by using the above lattice constants as given in Table II, together with one calculated from the Elser-Henley model.[22]

It is known that the icosahedral QC's Al-Pd-Re shows the highest electrical resistivity in icosahedral QC's,[23,24] and that

TABLE II. The structural vacancy densities for icosahedral QC $Al_{70.7}Pd_{21.34}Re_{7.96}$, $Al_{71.5}Pd_{20.3}Mn_{8.2}$, and $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$. The value of $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$ is calculated from the Elser-Henyley model (Ref. 22).

| Alloy | Structural vacancy density ($cm^{-3}$) |
|---|---|
| I-$Al_{70.7}Pd_{21.34}Re_{7.96}$ | $7.7\times 10^{20}$ |
| I-$Al_{71.5}Pd_{20.3}Mn_{8.2}$ | $5.0\times 10^{20}$ |
| $\alpha$-$Al_{68.31}Mn_{21.21}Si_{10.48}$ | $9.8\times 10^{20}$ |

its resistivity is much higher than the one in icosahedral QC's Al-Pd-Mn. However, the conduction mechanism of icosahedral QC's Al-Pd-Re is not fully established. Kimura *et al.*[12] suggested that the extremely low conductivity of Al-based icosahedral QC's originates from the covalent bonding nature of the 12-atom Al icosahedron occurring locally due to generation of vacancy at the center site of the icosahedron, and that the 12-atom icosahedron can be stable under covalent bonding. The present experimental results of the stable Al-based icosahedral QC's support strongly this picture.

Recently, Hiraga *et al.*[25] have discussed the atom cluster arrangements in 1/1 cubic approximant phase ($\alpha$-Al-Pd-Mn-Si) and 2/1 cubic approximant phase ($\beta$-Al-Pd-Mn-Si), and proposed the structure models of *F*-type icosahedral QC Al-Pd-Mn on the basis of the structure of $\alpha$-Al-Pd-Mn-Si and $\beta$-Al-Pd-Mn-Si. The structure models of icosahedral QC Al-Pd-Mn derived from these phases may reproduce well a diffraction pattern of icosahedral QC Al-Pd-Mn, however, the cluster model of icosahedral QC Al-Pd-Mn derived from the structure of $\alpha$-Al-Pd-Mn, which has no vacant sites, is obviously contradictory to our present conclusion. We propose that any structure modeling of QC Al-Pd-Mn or Al-Pd-Re should consider the presence of structural vacancies of $5-8\times 10^{20}\,cm^{-3}$ (concentration of about 1%).

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