

# Surface layer self diffusion in icosahedral Al–Pd–Mn quasicrystals

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## Abstract

The self diffusion of Mn and Pd in a single grain icosahedral Al<sub>69.9</sub>Pd<sub>20.5</sub>Mn<sub>9.6</sub> quasicrystal has been determined by low energy ion scattering (LEIS). The diffusion was determined by depositing different elements (Pd, Mn) on the surface and measuring the rate of change in surface composition as a function of temperature by LEIS. The surface composition was monitored over the temperature range of 355–575 K for Mn and 440–745 K for Pd and compared to model calculations to allow the activation energy for diffusion to be determined. Activation energies of  $0.20 \pm 0.01$  eV for Mn and  $0.64 \pm 0.03$  eV for Pd have then been measured for self diffusion in i-Al–Pd–Mn, respectively. No deviation from Arrhenius behavior was detected in the temperature range covered by the present experiments. From the low values of activation energy we propose that this range of diffusion is phason related, reflecting the specific nature of the icosahedral structure.

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## 1. Introduction

Quasicrystals (QCs) are well-ordered, but aperiodic, intermetallics. They exhibit some interesting material properties including low friction, low adhesion, high hardness and high wear resistance [1–5]. In spite of the large number of investigations performed on quasicrystals since their discovery [6,7] in 1982, little work has been devoted to the atomic transport in these materials [8–19], though an understanding of diffusion is of great interest for both their production and technological application. In the first theoretical study of diffusion in QCs, Kalugin and Katz [20] suggested that diffusive motion may be mediated by a process using phasons, which are defects that occur where the quasicrystalline matching rules are violated. Matching rules are a specific geometric property of the QC structures which have to be obeyed during building of the QC lattice from basic blocks into a perfect quasilattice [21]. These

authors proposed that phason controlled diffusion should dominate over diffusion via vacancies and should show up as a non-Arrhenius type temperature dependence of the diffusion rate if measured over a very broad temperature range. The first experimental diffusion study in a quasicrystalline material was done by Böttiger et al. [22] using Rutherford-backscattering spectrometry. This study was followed by a series of experiments using different techniques [8–19], in which values of activation energy and diffusion coefficients were measured. Bluher et al. [12] reviewed the diffusion results and concluded that the pre-exponential factors in the temperature dependence of the diffusivity,  $D$ , for all bulk-diffusion coefficients were  $10^{-4}$  m<sup>2</sup> s<sup>-1</sup> or larger with activation energies ranging between 1.5 and 2.6 eV, and that the diffusion was vacancy mediated diffusion. However, Bluher et al. also reported low temperature (<450 °C) radiotracer investigations of Pd and Au diffusion which gave very small activation energies. Kalugin and Katz [20] concluded that the Pd is diffusing via a phason-assisted mechanism, because the activation energy is low for the phason method compared to bulk vacancy assisted diffusion.

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This paper presents the results of an experimental study of Mn and Pd diffusion through the surface layer in icosahedral  $\text{Al}_{69.9}\text{Pd}_{20.5}\text{Mn}_{9.6}$  by low energy ion scattering (LEIS). LEIS using noble gas ions is a quantitative technique that is uniquely sensitive to the elemental composition of the topmost surface layers [23]. This is due to a combination of shadowing of subsurface layers and more importantly a high neutralization probability of incident and exiting noble gas ions.

In this experiment a submonolayer of a metal (Mn, Pd) was deposited on a clean, well annealed surface and the composition of the surface layer was monitored as a function of time at a fixed temperature. The sample was then cleaned, a new layer deposited and then the experiment repeated at a higher temperature. The concentration,  $C(x, t)$ , of a diffusing thin layer is given by

$$C(x, t) = C_o / \sqrt{(\pi Dt)} \exp(-x^2/4Dt)$$

where  $D$  is the diffusion constant and  $C_o$  is the total material comprising the thin film.

Our analysis is based on this equation and the approach described by Luscher et al. [19] who recently reported the diffusion rate of Al into the surface of an Al–Pd–Mn quasicrystal. There are two important differences between their work and that reported here. The first is that they used Auger electron spectroscopy (AES) which has an effective sampling depth of 0.8 nm (or about three atomic layers) while in this study low energy ion scattering has been employed as it has a sampling depth of typically one atomic layer. Furthermore Luscher et al. deposited 2–3 nm of Al before monitoring its diffusion, whereas in this study typically half an atomic layer of the diffusing material (Mn and Pd) was deposited. The analysis used in this study is similar to that of Luscher et al. who related their loss of deposited material to the diffusion constant.

Consider an element (Mn or Pd) which has an equilibrium concentration of  $C_a$  on the clean, annealed surface. If an additional amount,  $C_o$ , is deposited on the surface then at the start of a measurement there will be a surface concentration of  $C_a + C_o$  which will reduce asymptotically to  $C_a$  over time through diffusion. The quantity measured on the surface at any time during this process will be  $C_a + C(0, t)$  and it can be related to the diffusion coefficient by

$$C_o/C(0, t) = \sqrt{\pi Dt}$$

which, by the proportionality between concentration and measured yield, provides the relationship

$$Y_o/Y_t = \sqrt{\pi Dt}$$

where  $Y$  is the measured scattered ion yield scattered from the surface atoms of the element under investigation.

## 2. Experiment

The experiments were performed in a Leybold–Heraeus UHV (base pressure  $10^{-10}$  mbar) ion scattering system with

monoenergetic 2 keV  $\text{He}^+$ . The ions were generated using a Colutron ion source and were mass analysed prior to hitting the surface at  $30^\circ$  incidence. The scattering angle was  $60^\circ$  in order to obtain high yield with good mass resolution. A single grain sample of i-Al–Pd–Mn, with a nominal composition of  $\text{Al}_{72}\text{Pd}_{19.5}\text{Mn}_{8.5}$ , was grown at the Ames Laboratory by the Bridgman method. The sample was oriented with the surface perpendicular to the fivefold axis. SEM-EDS determined the sample composition to be  $\text{Al}_{69.9}\text{Pd}_{20.5}\text{Mn}_{9.6}$ . The sample was initially cleaned in vacuum by cycles of  $\text{He}^+$  sputtering and annealing. An annealing temperature of 450 K was used for the first cleaning cycle. This was increased by 50 K after each cycle until a final annealing temperature of 800 K was used. The sample was heated from the rear using a tungsten filament for temperatures below 700 K and an electron beam for temperatures above 700 K. After the cleaning procedure and prior to each experiment, the sample was sputtered using  $\text{He}^+$  and annealed at 800 K for one minute.

Mn was evaporated onto the annealed sample using an thermal evaporator made from an alumina crucible with a tungsten filament wrapped around it. Pd was deposited using an Oxford scientific evaporator (OS-vap) using e-beam heating.

## 3. Results

The LEIS spectra of 1.9 keV He ions scattered off the surface before cleaning (Fig. 1) initially reveal a surface with a strong surface concentration of Al (1620 eV), and a much weaker Pd peak (1820 eV). As well there is a distinct peak (1460 eV) which is a surface oxygen contamination. After cleaning, the LEIS yielded an energy spectrum with no clear peak corresponding to scattering from or recoil of oxygen. This is indicative of a surface with less than 1% oxygen coverage. As well there was a strong sharp Al peak, a significant Pd peak and a weak Mn peak (1720 eV). The surface coverage of Mn increased significantly under room temperature ion bombardment.

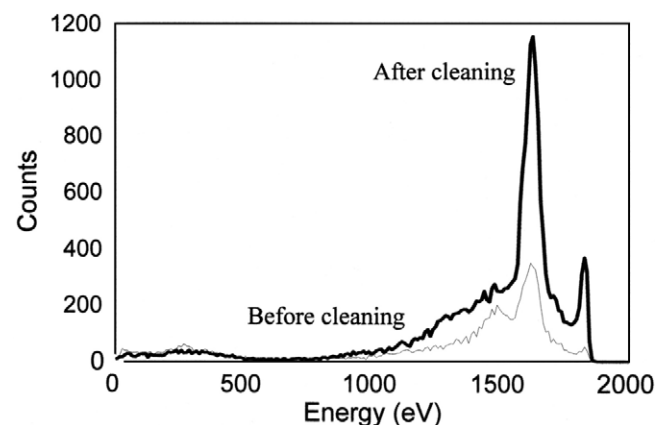


Fig. 1. LEIS energy spectrum of 2 keV  $\text{He}^+$  scattered from an Al–Pd–Mn quasicrystal before (dashed) and after (solid) cleaning. The angle of incidence to the surface was  $30^\circ$  and the scattering angle was  $60^\circ$ .

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