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Electronic and atomic disorder in icosahedral AlPdRe

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Abstract

Relations between electronic and atomic disorder of i-AlPdRe have been investigated by studies of neutron irradiated and annealed samples. The advantage with this technique is that a single sample can be monitored over a significant range of varying electronic properties, without concern for any influence of varying impurities. X-ray diffraction, the electrical resistivity and its temperature dependence, and the magnetoresistance are studied. The results show that annealings of an irradiated sample lead to improvement of the atomic order, as reflected in increased intensities of the x-ray diffraction peaks, while electronic properties change in the direction of increasing electronic disorder towards a metal–insulator transition. The observed relation in quasicrystals that improved atomic structure is associated with stronger anomalies in transport properties is thus also seen in i-AlPdRe. In particular, the variation of the diffusion constant in the region of small values of the resistivity is found to be similar for annealed polygrain samples and for single grain samples with varying Pd concentration, as evaluated from literature data, indicating a similar development of electronic disorder in both sets of samples. However, the problem remains as to why the resistivity is small in single grain samples which are atomically well-ordered. The possibility of a strong sensitivity to concentration differences is pointed out.

1. Background

Icosahedral quasicrystals display a number of remarkable electronic transport properties. One example is the large range of resistivities, ρ, with values at 4.2 K from below 100 $\mu\Omega$ cm to above 1 Ω cm. Another striking feature is the often strongly negative $\mathrm{d}\rho/\mathrm{d}T$, with an average temperature dependence

$$R = \frac{\rho(4.2\ \mathrm{K})}{\rho(295\ \mathrm{K})}, \tag{1}$$

which can reach large values not previously encountered in metallic alloys. A third example is the surprising observation that improved sample quality can lead to an increase of ρ and R, in contrast to the decrease in resistivity which usually accompanies removal of defects in simple crystalline alloys.

Icosahedral (i) AlPdRe is particularly challenging. In this case polygrain samples of the same nominal composition, which are phase pure in standard x-ray diffraction, can be prepared by different heat treatments over a range of low temperature resistivities varying by several orders of magnitude and with R-values from about 2–300. In contrast, single grain samples have low temperature ρ-values of only a few mΩ cm and small R-values in the range 1–2 [1, 2]. Single grain samples are expected to be more perfect than polygrain samples and it may thus appear that the anomalous relation between sample quality and resistivity mentioned above is violated for i-AlPdRe. As will be seen, however, this is not necessarily the case. The large range of R-values in polygrain samples corresponds to a wide range of different physical properties from weakly disordered metals to insulators. It is poorly understood which underlying physical change can explain this variation. This fact, combined with the metallurgical difficulties, have led to continued concerns for more than 10 years of studies of i-AlPdRe that the observed or assumed second phases would influence the physical results [3, 4].

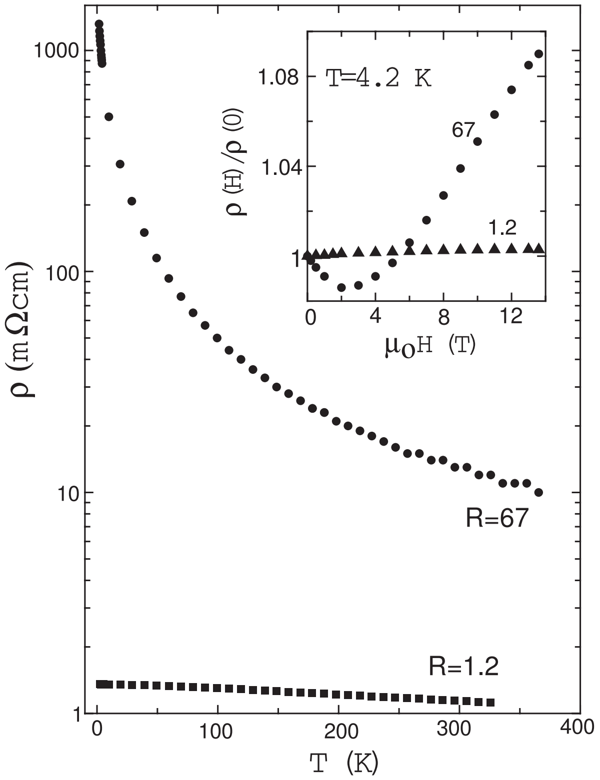

Figure 1. Range of variation of $\rho(T)$ (main panel) and the magnetoresistance (inset) under high energy neutron irradiation of an original i-AlPdRe sample with $R = 67$. The variation of $\rho(T)$ and R covers (on logarithmic scale) most of the change of these properties which can be obtained in i-AlPdRe. Here this variation is achieved in a single sample. When the neutron dose increases from 0 to 6.8×10^{19} cm^{-2}, ρ and $|\mathrm{d}\rho/\mathrm{d}T|$ decrease monotonously, while $\Delta\rho(H)/\rho(0)$ at 4.2 K and 2 T first changes sign and the magnetoresistance increases to about 1.01 at $R \approx 13$ and then decreases with further increasing dose [5].

Experiments with high energy neutron irradiation of a polygrained i-AlPdRe sample of large resistivity and R-value have clarified some of these problems [5]. With increasing neutron irradiation dose a continuous decrease of ρ and R was found. X-ray diffraction experiments verified that only quasicrystalline peaks were observed of strengths which decreased with increasing neutron dose. Starting with an $R = 67$ sample, the effect on R of irradiation saturated at about 7×10^{19} neutrons cm^{-2} where R had decreased to 1.2. This value is somewhat larger than the upper limit of R in amorphous metals. The experiment relates changing electrical properties to changes of the icosahedral phase in one sample of constant impurity concentration, and therefore supports the contention that the physical results are not influenced by impurity phases.

Figure 1 illustrates $\rho(T)$ and magnetoresistance of one i-AlPdRe sample before neutron irradiation and after a maximum irradiation dose. At He-temperatures $\rho(T)$ changes by a factor $\sim$1000, and the magnetoresistance at 4.2 K, expressed as $\Delta\rho/\rho = [\rho(13\text{ T})/\rho(0)] - 1$, varies from 9% to 0.3%. While there is no feature in the results for $\rho\ (T, R)$, which would signal an insulator–metal transition with decreasing R, the magnetoresistance displays such a transition with a typical insulating behavior in Efros–Shklovski variable range hopping at large R, and weak localization and enhanced electron–electron interactions at small R. The metal–insulator transition, MIT, is estimated to occur at about $R \approx 20$. It is this large range of variation of physical properties which is the challenge to understand.

This problem is at present too difficult. Our paper aims at investigating some aspects of related questions by studies on the metallic side of the MIT. Annealing experiments on a strongly neutron irradiated sample have been performed. After a brief description of the experiments, the results are described in section 3, and discussed in section 4. These experiments show that the standard quasicrystalline property of an *increase* of ρ(4.2 K) and R with improved quasicrystalline quality is obeyed in studies on one single (polygrained) sample. This reinforces the necessity to distinguish between electronic and atomic disorder in quasicrystals. The importance of electronic disorder is illustrated in section 4.2 by both the increase of the inverse diffusion constant, D^{-1}, and of the inelastic scattering rate as a function of R. In section 4.3 it is shown that the relation between D^{-1} and R is similar for annealed polygrain samples and single grain samples, which suggests that electronic disorder also accounts for changing electronic properties in single grain samples. The problem of why single grain samples have small ρ(4.2 K) and R is mentioned in section 4.4. The possibility of a strong sensitivity in the electronic transport properties of i-AlPdRe to small changes of chemical concentration is pointed out.

2. Experiments

Samples of nominal composition $Al_{70.5}Pd_{21}Re_{8.5}$ were prepared by arc-melting and successive annealings as earlier described [6]. For the annealing experiments, a sample with $R = 67$ was selected. It had been exposed to a series of irradiations with increasing high energy neutron doses. The total range of variation of $\rho(T)$ and the low temperature magnetoresistance obtained in this process is shown in figure 1.

After an irradiation with 6.8×10^{19} neutrons cm^{-2} the sample was annealed for 20 min at a series of low temperatures T_a, which were increased from 100 °C in steps of 50 °C. After each step $\rho(T)$ was measured from He-temperatures to about 330 K and the magnetoresistance was measured at 4.2 K up to $\mu_0 H = 13.6$ T. After each such measurement, contacts were carefully removed before the next annealing step. At a few steps x-ray diffraction patterns were taken with Cu Kα radiation. The variation of R in the annealing experiments is limited to the low R metallic region. Previous results [6] for metallic samples have also been used to discuss the transport results in this region of R.

3. Results of annealing experiments

All peaks observed in the x-ray diffraction patterns could be indexed on the icosahedral AlPdRe phase. It has been reported previously that with increasing irradiation dose, the height of the diffraction peaks decreased, while peak widths remained unchanged within experimental resolution [5]. We

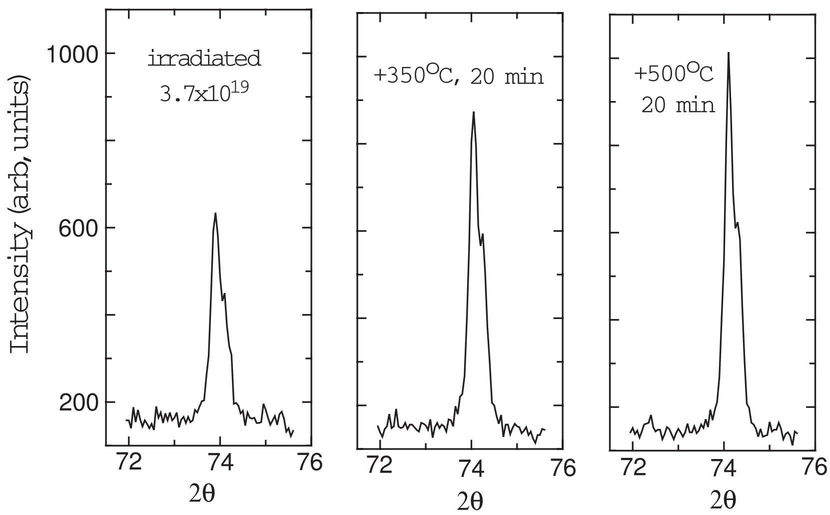

Figure 2. The 52/84 peak of i-AlPdRe after irradiation with 3.7×10^{19} neutrons cm^{-2} and after subsequent annealings for 20 min at 350 and 500 °C. Cu Kα irradiation was used in x-ray diffraction. The intensity of the peak increases with annealing temperature.

found that the peak heights after annealings increased, while still no change in peak half widths could be detected. The increase in peak height with annealing temperature is shown in figure 2 for the 52/84 reflection[5]. This peak occurs at a relatively large scattering angle, demonstrating the long range icosahedral atomic order after irradiation. Its growth with annealing is also seen. A similar increase of intensity was observed for all peaks.

This recovery of the icosahedral phase occurs at low temperatures, i.e. at substantially lower temperatures than used in the annealings in the sample preparation procedures. Closer inspection of the figure also shows that the Bragg angle increases slightly with increasing annealing temperature. At 500 °C, 2θ of the 52/84 peak has increased by 0.2°, corresponding to a decrease of the six-dimensional lattice parameter of about 0.4%.

The electrical resistivity was found to increase with the annealing temperature T_a. Figure 3 illustrates ρ as a function of T_a at 4.2 K and room temperature. On the scale of the total change of the resistivity with irradiation in figure 1, the change of ρ with T_a is tiny. One should probably prolong the lower temperature annealing times to cover a larger region of changes in ρ-values. The conclusion from figure 3 is nevertheless that reversible electrical properties over a small range of ρ-values can be monitored in an i-AlPdRe sample by irradiation and annealing. It can be seen that the difference between the two curves in the main panel of figure 3 increases slightly with increasing T_a. This corresponds to an increase of the resistance ratio R with T_a, illustrated in the inset. This growth of R can thus be associated with a recovery of the icosahedral phase.

The magnetoresistance is illustrated in figure 4. Data at 4.2 K and 2 T are shown as a function of log R. The present results for annealed samples, shown by open circles, have been plotted onto the lower R-part of a previously published graph for as-made and for irradiated samples (figure 5 of [5]).

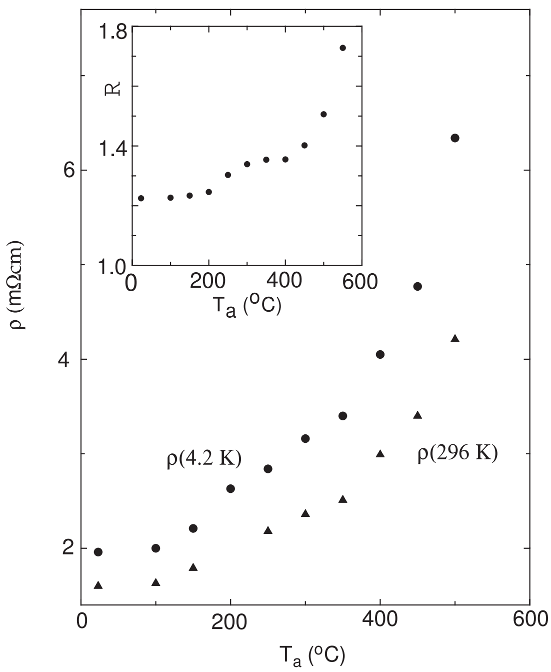

Figure 3. The resistivity of i-AlPdRe irradiated with 3.7×10^{19} neutrons cm^{-2} versus T_a at 4.2 and 296 K. Inset: the resistance ratio R versus T_a.

Samples with R below about 4 in that graph are shown by filled circles in figure 4.

An estimated experimental error of about 0.1% is shown for one datum. It is large on the scale of $\rho(H)/\rho(0)$ and accounts for most of the scatter in the data[6]. It can be inferred from figure 4 that the trend of a decrease of $\rho(H)/\rho(0)$ versus R with increasing irradiation dose is reversed by increased annealing temperatures. The magnetoresistance versus R

[5] 52/84 corresponds to the 6D fcc reflection $(h, h', k, k', l, l') = (4, 6, 0, 0, 0, 0)$.

[6] The only case of an exceptional datum was at (1.354, 1.0061). It is probably an error and has been omitted.

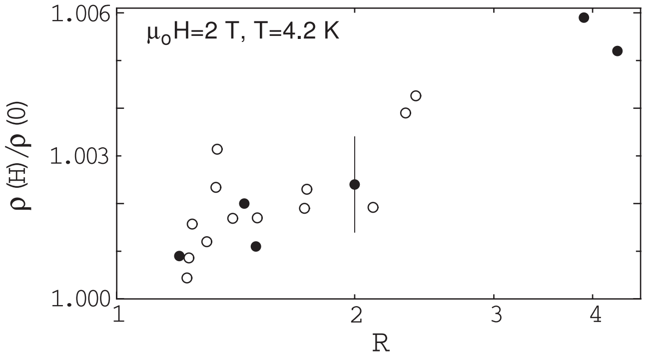

Figure 4. The magnetoresistance versus log R. Filled samples are from the low R region in figure 5 of [5], and include as-made samples and samples irradiated with smaller dose. Open symbols from the present annealing experiments are seen to roughly follow the same trend of $\rho(H)/\rho(0)$ versus R.

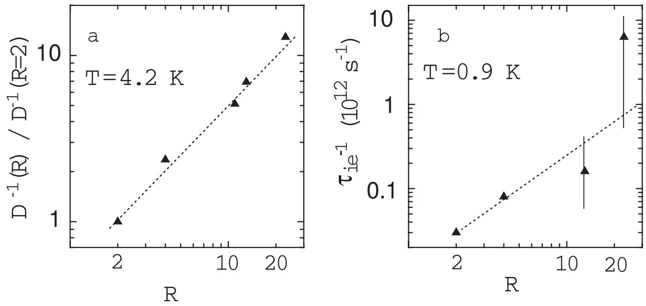

Figure 5. Illustrations of electronic disorder in i-AlPdRe. (a) The inverse of the diffusion constant normalized to its value at $R = 2$ versus R. D was estimated from equation (3). The straight line is an empirical description of data. It gives an upper limit for the increase of the elastic scattering rate when the MIT is approached. (b) The inelastic scattering rate as a function of R. τ_{ie} was estimated from the magnetoresistance [6].

now qualitatively describes the same relation in the opposite direction.

The results can be compared with transport results for SmB_6 [7]. SmB_6 has a narrow band gap, as in calculations for ideal i-AlPdRe [8]. ρ increases strongly in SmB_6 for decreasing temperature below about 10 K. After neutron irradiation ρ at low T decreases by a factor ∼500, and SmB_6 passes through an insulator–metal transition. Also, after annealings ρ partly recovered. These similarities are interesting.

4. Consequences and discussions

4.1. Intrinsic properties of the icosahedral phase

The present annealing experiments put on firm ground the evidence from neutron irradiation studies that intrinsic electronic properties of the icosahedral phase are measured. The evidence for this conclusion is briefly summarized. One sample was driven by neutron irradiations from an insulating state towards a weakly disordered metal [5] and has now been reversibly transformed in the opposite direction over a small range of R-values by low temperature annealings. These changes are related to changes of the icosahedral phase as observed by the decreasing intensities of the x-ray peaks for increasing neutron irradiation and the increased intensities of these peaks for increasing annealing temperatures. No additional peaks are observed in x-ray diffraction. Impurities from irradiation products are negligible compared to the impurity level of the starting materials. Phase transformations during irradiation are unlikely. The annealing temperatures are too low for phase transformations such as the growth of an amorphous phase. The impurity level in the sample can therefore be considered to be constant during both the irradiation and the annealing experiments.

4.2. Distinction between electronic and atomic disorder

We thus find a relation between structure and electronic properties for i-AlPdRe which apparently is similar to that observed in other quasicrystals; improved icosahedral order is associated with larger low temperature resistivities and larger values of R. Larger resistivities by themselves signal electronic disorder. It is hence necessary to distinguish in quasicrystals between electronic and atomic disorder.

Electronic disorder can be quantified by the parameter

$$\Im = \frac{\hbar}{\tau \varepsilon_F}, \qquad (2)$$

where $\hbar$ is Planck's constant$/2\pi$, τ is the elastic scattering time and ε_F is the Fermi energy. Equation (2) shows that $\Im$ is related to the resistivity, and increases for an increasing ρ. Atomic disorder in the present experiments could be quantified, e.g. by a decreasing volume of the coherent icosahedral phase.

We illustrate the increasing electronic disorder in our well-ordered icosahedral samples when approaching the metal–insulator transition from the metallic state in i-AlPdRe in two ways; (i) from the elastic scattering time and (ii) from the inelastic scattering time.

(i) The conductivity is Boltzmann-like in the metallic regime with corrections from weak localization and enhanced electron–electron interactions. $\rho = [e^2 N(\varepsilon_F) D]^{-1}$, where $D(= \tau v_F^2/3)$ is the diffusion constant, $N(\varepsilon_F)$ is the density of states, and v_F is the Fermi velocity. The temperature dependence of ρ is dominated by $\tau(T)$ in D. Both $N(\varepsilon_F)$ and D are affected by an increasing R when the MIT is approached. However, one can eliminate $N(\varepsilon_F)$ by using the empirical relation $N(\varepsilon_F) \sim 1/\sqrt{\rho(295\ \mathrm{K})}$ obeyed for a large range of quasicrystals [9, 10]. Neglecting any temperature dependence of $N(\varepsilon_F)$ one then finds

$$D(4.2\ \mathrm{K}) \sim \frac{\sqrt{\rho(295\ \mathrm{K})}}{\rho(4.2\ \mathrm{K})} = \frac{1}{\sqrt{\rho(4.2\ \mathrm{K})R}} \qquad (3)$$

giving a convenient estimate of D from the easily accessible parameters $\rho(4.2\ \mathrm{K})$ and R.

Figure 5(a) illustrates the increase of D^{-1} normalized to its value at $R = 2$ when the MIT is approached. This increase may be due to a combination of an increasing elastic scattering rate τ^{-1} and a decreasing Fermi velocity v_F. The straight line is an empirical summary of data and has a slope close to 1, suggesting that D^{-1} is linear in R in this region of R-values.

This line represents an upper bound for the rate of increase of τ^{-1} when approaching the MIT.

(ii) The inelastic scattering rate τ_{ie}^{-1} in the metallic regime can be evaluated from the magnetoresistance [6]. The uncertainty of these results increases when the MIT is approached and analyses of the magnetoresistance in terms of weak localization theories break down. Figure 5(b) shows results for τ_{ie}^{-1} at 0.9 K. Each datum was obtained by averaging between different analyses [6]. τ_{ie}^{-1} increases strongly when approaching the MIT. In fact, the results in figure 5 indicate that τ_{ie}^{-1} increases more strongly than τ^{-1}, eventually leading to break down of the condition $\tau_{ie} \gg \tau$, necessary for the observation of weak localization effects.

The results in figures 5(a) and (b) show two independent sources of confirmation that electronic disorder increases with increasing R in i-AlPdRe. In panel (a) this is evident from the inferred increase of τ^{-1} and the definition of the disorder parameter, equation (2). The conclusion from panel (b) is in agreement with general results for disordered metals and semiconductors which show that the dephasing rate increases with the resistivity [11].

4.3. Electronic disorder accounts for varying properties also in single grain samples

Atomic disorder can be of different types and lacks the immediate connection to a simply measurable sample property, such as the electrical resistivity for quantifying electronic disorder. In early results for i-AlCuFe [12], for example, samples in the as-quenched condition showed broad icosahedral x-ray diffraction peaks and an impurity peak, and the increased ρ-and R-values upon annealing were accompanied by narrowing peak widths and vanishing impurity phase. In the present samples impurity peaks do not appear upon irradiation, and peak widths do not change under irradiation and annealing. Improved icosahedral order with annealing is in this case reflected in the increasing peak heights.

The range of R-values obtained for single grain samples [1, 2] and by annealings of a heavily irradiated polygrain sample are similar, and the diffusion constants can be directly compared using equation (3). This is shown in figure 6. The filled triangles are taken from figure 5(a), and the straight line in figure 6 is an extrapolation of the line in figure 5(a) to the region $R < 2$. Several annealed polygrained samples with T_a up to 500 °C (open circles) and single grain samples (filled square and circles) are shown. The bars on the filled circle at $R = 1.67$ indicate the area in the (D^{-1}, R)-plane which was accessed by various annealings at fixed chemical composition in [2]. Each filled circle in the figure represents an average value obtained from such different heat treatments.

The relation between D^{-1} and R in figure 6 is similar for three different sets of i-AlPdRe samples; i.e. as-made polygrain i-AlPdRe, a heavily irradiated polygrained sample subsequently annealed in various steps, and single grain samples. The common ground for the results in figure 6 is an

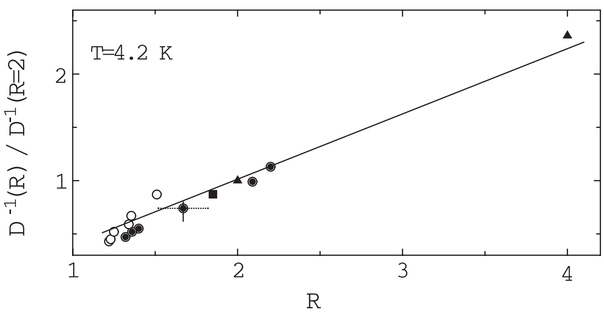

Figure 6. Electronic disorder of annealed polygrained and single grained samples in the small R-region. O; present annealed samples, ▲; data at $R = 2$ and 4 from figure 5. ■; single grain sample from [1]. ●; data for single grain samples from [2]. Average values for each Pd concentration are shown[7]. The bars around one sample indicate the variation of R and ρ between different heat treatments at constant Pd concentration. The straight line was taken from figure 5(a) and extrapolated to the region $R < 2$.

increasing electronic disorder with increasing R at a rate which is independent of the details of the varying atomic order. For polygrain samples it is established that electronic properties are those of a weakly disordered electron gas [6]. It can therefore be conjectured from figure 6 that the single grain samples are similarly weakly disordered.

The partial destruction of atomic order by neutron irradiation is apparently a much weaker distortion than that obtained in quenched i-AlCuFe [12]. In the present case a pure icosahedral phase is observed after all irradiations and annealings and it is the peak intensities not the peak widths that are affected. One can perhaps ascribe this to the volume of preserved coherent icosahedral regions which decrease under irradiation and increase with annealing.

Why are the electronic properties quite similar for a range of samples of different atomic order in figure 6? The coherence length L of the icosahedral phase for a single grain i-AlPdRe sample was estimated to be $L \approx 800$ Å [2], and for a neutron irradiated polygrain sample similar estimates of the width of the main x-ray diffraction peak indicated a value of L of order half that value [5]. This can be compared with an upper limit for the longest electronic length scale, i.e. the inelastic scattering length L_{ie}. Using $L_{ie}^2 = D\tau_{ie}/3$ one can take $D = 5.5 \times 10^{-6}$ m^2 s^{-1} at $R = 4$ from [13], and multiply by a factor 4, corresponding to the ratio of D at $R = 1.2$ and 4 in figure 6. Extrapolation in figure 5(b) of $\tau_{ie}^{-1}(T)$ to the same region of R gives $\tau_{ie} \approx 5 \times 10^{-11}$ s. An upper limit of L_{ie} is therefore $L_{ie}^{max} \approx 200$ Å.

We thus find that $L_{ie} < L$ over the full measurement range in R and T. The icosahedral environment is intact from the point of view of electronic events both for heavily irradiated and annealed polygrain samples and for single grain i-AlPdRe. It seems that once a certain level of atomic order has been achieved, further improvements do not affect electronic properties.

[7] In [2] the definition of R was $\rho(1.8\text{ K})/\rho(300\text{ K})$. The difference from equation (1) in the text is negligible, since $|d\rho/dT|$ is quite small in this region of R.

4.4. Major open problems

However, some of the most important questions are not solved. Relevant changes in atomic order with neutron irradiation and annealing have not been identified, except for the finding that they are not dramatic, since long range icosahedral order is preserved. A second phase problem is not a likely scenario as discussed above. A decrease of $\rho(4\ \text{K})$ and R with a disturbance from energetic neutrons impinging on the atoms in polygrain samples as well as a (partial) recovery with annealing is in line with the well-known properties for quasicrystals that increased atomic disorder is accompanied by decreased electronic disorder. However, it appears there is no casual connection between these two types of disorder. If we ask conversely why the presumably increased atomic order of single grain i-AlPdRe would not lead to increased electronic disorder and a larger ρ, we have no answer.

An unexplored possibility to resolving some of these conundrums is differences in chemical composition. The properties of quasicrystals can be extremely sensitive to small changes in electron concentration, since the scattering vectors are sufficiently numerous to make up a pseudo-Brillouin zone which is almost spherical. Minor changes of the charge may then cause strong changes in contact between Fermi surface and pseudo-zone boundaries and consequently strong changes of electronic properties. For example, a change of Fe concentration in i-AlCuFe of below 0.05 at.%, corresponding to a change of the electron per atom ratio of 0.1%, was found to cause a dramatic jump and sign change in the Hall coefficient [14]. A strong sensitivity to small concentration variations in electronic properties in i-AlPdRe was inferred from NMR experiments [15]. In the single grain i-AlPdRe experiments [2] the Pd concentration was varied. In this case no signs were found of any dramatic change of electronic properties. However, with the large steps of 1–2 at.% Pd between different samples, this experiment does not provide evidence for the absence of a strong sensitivity to concentration variations. Furthermore, in the i-AlPdRe we studied the nominal Re concentration was 8.5% while in [2] it was 7 at.%.

5. Brief summary

Annealing experiments have been carried out on a strongly neutron irradiated i-AlPdRe sample. This experiment, together with neutron irradiation experiments, are so far the only methods to monitor the electronic properties of polygrain samples in the low R-region characteristic for single grain samples. The results from annealings show a reverse trend from the irradiation experiments; for increasing T_a the x-ray diffraction peak heights increase and $\rho(4.2\ \text{K})$, R, and $\rho(H)/\rho(0)$ also increase. Measured properties are intrinsic to the icosahedral phase since a single sample can be studied over the full range of variable electronic properties of i-AlPdRe and reversibly over a small range of R values by different annealings. Also for i-AlPdRe one can thus observe the usual quasicrystalline correlation that improved sample quality leads to increased ρ and R. Such a relation has long been expected but hitherto not experimentally confirmed.

The elastic and inelastic scattering times were studied in the metallic regime at small R. The increase of the inverse diffusion constant with increasing R is similar in polygrain as-made samples, irradiated and annealed polygrained samples, and single grain samples. This result underlines the usefulness of R as a single parameter characterizing the electronic state of i-AlPdRe.

The apparent contradiction that single grain i-AlPdRe shows evidence of weak electronic disorder rather than the expected strong disorder has been discussed. A strong sensitivity to small concentration changes, not yet verified for i-AlPdRe, could possibly provide an explanation.

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