



# Trapped muon in heavily dislocated palladium

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## Abstract

Using the zero magnetic field technique, muon spin relaxation has been measured in dislocated and non-dislocated palladium containing hydrogen. The technique has proved to be a sensitive way of demonstrating muon trapping on dislocations. In the non-dislocated palladium, no depolarization of the muon was observed owing to the low concentration of hydrogen, whereas in the dislocated sample at the same hydrogen concentration, a clear depolarization was observed, indicating that the muon must be trapped on dislocations adjacent to trapped protons. The depolarization was observed to increase as the temperature was reduced, consistent with tighter trapping of hydrogen around dislocation.

*Keywords:* Palladium; Dislocation; Muon spectroscopy

## 1. Introduction

The study of dislocations by the measurement of small angle neutron scattering from the resulting lattice distortion is well established, having been first predicted (for X-rays) by Atkinson and Hirsch [1]. These authors showed that we would expect to see a  $Q^{-3}$  dependence from edge dislocations. It is also well established that H or D will be trapped near a dislocation, the trapping energy being given by  $\sigma V_H$  where  $\sigma$  is the local stress and  $V_H$  is the partial molar volume of H(D) in the lattice and  $\sigma$  varies as the reciprocal distance from the dislocation, [2,3]. In metal hydrides the distribution of H(D) on appropriate interstitial sites near the dislocation is actually given by a Fermi–Dirac distribution because there can only be one H(D) atom on each site.

In this paper we describe qualitative muon spectroscopy measurements designed to demonstrate that muons are trapped on dislocations. We believe this to be the first time that muon trapping on a dislocation in a metal–hydride has been demonstrated. The measurements were performed

using two palladium samples, chosen to have the same hydrogen concentration, in one of which a high density of dislocations was introduced by cycling through the  $\alpha/\beta$  phase. The other sample was well annealed with no dislocation present.

## 2. Experimental details

Palladium foils of 0.5 mm thickness and of 99.99% purity, from the Aldrich Chemical Company, were used during this experiment. Two foil samples were annealed at 900°C before being hydrogenated. The first sample was heavily dislocated by introducing hydrogen into it and cycling it through the  $\alpha/\beta$  phase at room temperature [4]. This is achieved by admitting a hydrogen concentration of  $[H]/[M]>0.65$  into the sample and then degassing it at room temperature under a vacuum of better than  $10^{-3}$  Pa for three days. This is to insure that most of the hydrogen not trapped in the dislocations is released. The concentration of the hydrogen left in the sample was calculated from the mass difference before and after hydrogenation. The sample was found to be in

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the  $\alpha$  phase and the concentration in the order of  $[H]/[M] \sim 0.01$ . The second sample was prepared in such a manner that it had the same hydrogen concentration as the first one but without going through the dislocation process. Furthermore, the hydrogenation was accomplished at high temperature to minimize the stress in the lattice and the dislocation density.

Muon spin resonance measurements were made on the EMU spectrometer at ISIS in the Rutherford Appleton Laboratory, UK, using longitudinal geometry and zero magnetic field. The measurements were made for both samples over the temperature range 5–200 K.

### 3. Results and discussion

Fig. 1 shows the muon spin relaxation obtained from the two samples at 5 K. It is clear from the figure that the relaxation rate in the dislocated sample is much the greater. In metal hydrides the principal source of the relaxation is the dipolar coupling between the muon and the nuclear spins of the metal and hydrogen atoms. The nuclear dipolar moment of Pd is essentially negligible in relation to the other moments and it is easy to show that the contribution from the metal is not observable. The dipolar interaction is also strongly dependent on the distance between the spins and, therefore, on the local configuration of the hydrogen atoms around the muon. To demonstrate the sensitivity of muon spin relaxation in detecting dislocations, the hydrogen content in the present experiment was chosen to be very low ( $[H]/[M] \sim 0.01$ ), within the concentration range of the hydrogen trapped in the dislocations. The intention was to ensure that the average muon–hydrogen distance in

the well annealed sample, a purely random solution, is too large to enable any significant depolarization of the muon.

In the dislocated sample, the second moment of the dipolar field distribution,  $\sigma_{ZF}^2$ , may be calculated by fitting a gaussian curve to the experimental data. The value of  $\sigma_{ZF}^2$  was found to be  $0.24 \mu\text{s}^{-1}$  at 5 K and this may be compared with the value of  $0.31 \mu\text{s}^{-1}$  found for  $\text{PdH}_{0.7}$  [5]. We may therefore conclude that the muon–hydrogen distances are similar in both these samples, i.e., is of the order of the average interatomic distance. Since the hydrogen concentration is so low, this is a strong indication that the hydrogen atoms and muons are grouped close together in the dislocated sample, the inference being that both decorate the dislocations. In contrast, the depolarization in the annealed sample is very weak, so much so that it is difficult to attach a precise value to it. Such a result is consistent with the low concentration of randomly distributed hydrogen atoms.

Fig. 2 shows the temperature dependence of the depolarization in the dislocated sample. These results are typical of muon diffusion and indicate the onset of motional narrowing at temperatures well below 150 K. However, the apparent diffusion rate in this case is much greater than that in other metal–hydrides and, in particular, is much greater than the diffusion rate in  $\text{PdH}_{0.7}$  [5], where motional narrowing begins to take place near 200 K. The difference can best be explained either by rapid diffusion along the decorated dislocations or by the release of the muon and/or the hydrogen from the dislocations as the thermal energy is increased.

In conclusion, these results demonstrate that hydrogen doping of a metal can be a very sensitive technique for detecting muon trapping, assuming that hydrogen and muon will both tend to trap at the same defects. Further measurements are planned for the near future to investigate

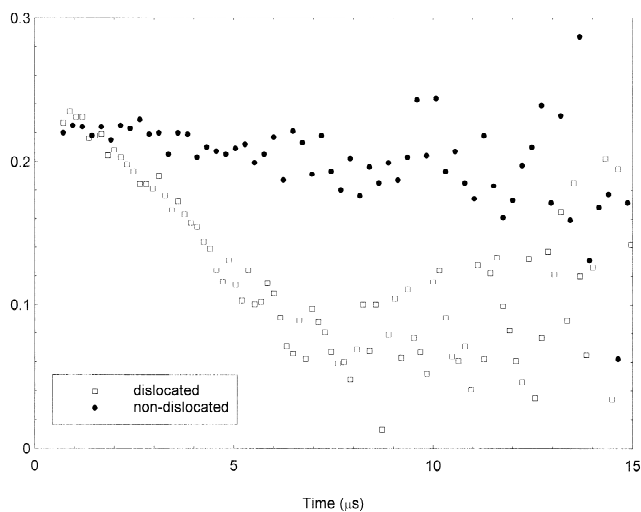


Fig. 1. Comparison of the muon spin relaxation,  $G_z(t)$  at  $T=5$  K, between the dislocated palladium hydride and the well annealed palladium hydride. It is very clear that the relaxation rate in the dislocated sample is much the greater.

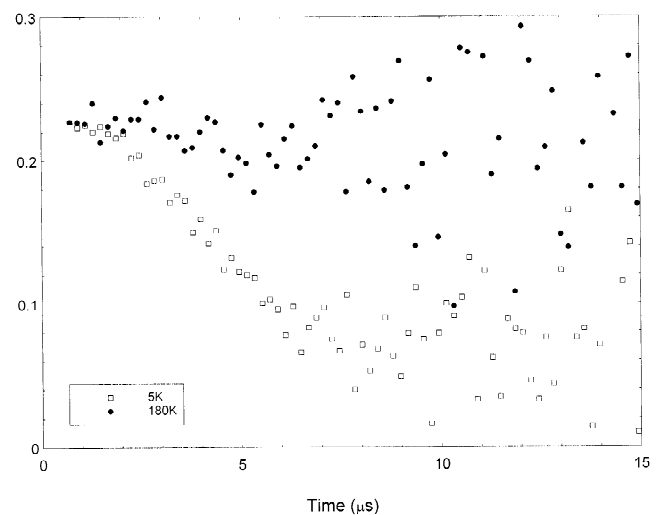


Fig. 2. Temperature dependence of the muon depolarization in the dislocated palladium containing interstitial hydrogen.

the origin of the rapid diffusion and also to establish the effect, if any, of altering the dislocation density.

## References

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