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Microstructure of ordered Pd–8at.%Y alloys

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Abstract

The microstructure of palladium–yttrium alloys has been investigated in this paper, the two alloys were the Pd–7.52at.%Y alloy and Pd–8.22at.%Y alloy, respectively. The Pd–Y alloy ingots were prepared by melting the yttrium powder and palladium powder in a vacuum induction furnace. The resultant alloys were homogenized at 950°C for 6 h. Then cold rolled to produce foil. Some of them were quenched into water from 900, 920 and 940°C, respectively. Finally given a vacuum anneal at 750°C for 40 min and furnace cooled to produce the material in long-range-ordered (LRO) condition. There was an inflection on the curve of electrical resistance vs. temperature, which indicates that the anomaly electrical resistance variation is due to an order–disorder transition. The results of XRD show that Pd_3Y typed LRO super-lattice existed in as-annealed samples. After annealing, the average grain size of Pd–8at.%Y alloys become smaller, the grains distribute unequally, and the grain scale for as-quenched and as-annealed samples was $750/mm^2$ and $760/mm^2$, respectively. Microstructure was studied by optical and transmission electron microscopy. Anti-phase domain boundary (APB) was found in the as-annealed alloys. The cell-like structures in the TEM photo were not complete.

Keywords: Pd–Y alloys; Microstructure; Order–disorder transition

1. Introduction

Materials of higher hydrogen-permeability, lower working temperature, which are used for nuclear reactors, are required on separating and purifying hydrogen. Nowadays, the hydrogen permeability of widely used Pd–25at.%Ag alloy is low, and can not meet the requirement. In order to improve it, particular emphasis has been placed on the Pd–RE alloys. The permeability of Pd–Y is 50% or so higher than that of Pd–25at.%Ag [1–3]. Because of their superior permeability, Pd–Y alloys are expected to replace Pd–Ag alloys in the future. The presence of ordered structure in the Pd–Y solid solution alloys, which improves the permeability was reported by Hughes et al. [1,2,4]. The permeability of Pd–8at.%Y alloys heated in H_2 is much higher than that of alloys unheated. It was the solution of hydrogen in the alloys that induced ordered structure, thus improving the permeability of the hydrogen diffusion membrane, which means the permeability of ordered alloys is over the disordered ones. In order to obtain higher permeability, it is helpful to make the diffusion membrane ordered by adjusting the heat treatment condition. So, the microstructure of two kinds of ordered Pd–Y alloys was studied in this paper.

2. Experimental procedure

The Pd–Y alloy ingots were prepared by melting palladium powder (purity, 99.98%) and yttrium powder (purity, 99.9%) in a vacuum induction furnace. The resultant alloys were covered by titanium foil, and homogenized in a vacuum annealing furnace at 950°C for 6 h. The materials were then cold rolled to produce foil, of which the thickness is 0.019 mm. Some of them were sealed in a silica tube, quenched into water from 900, 920 and 940°C, respectively. Simultaneously, the tube was broken and the surface oxide was brushed with sandpaper. It was finally vacuum annealed at 750°C for 40 min and furnace cooled to produce the materials in long-range-ordered (LRO) condition.

Microstructure was determined by a model XJG-05 optical and transmission electron microscope, respectively. The working voltage of model JEM-2000 TEM is 200.0 kV. Phase structures were detected by a D/max-RC200 total power Diffractometer. The electrical resistance R was

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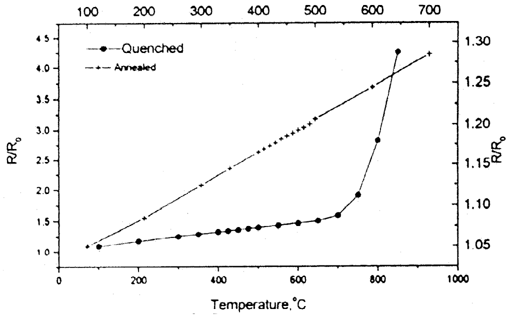

Fig. 1. The curve of R/R_0 vs. T.

measured at different temperatures and the corresponding electrical resistance at absolute zero R_0. R/R_0 was used as the longitudinal axis, temperature (T) as transverse axis, and a curve was made. From the inflection on the curve the order–disorder transition temperature can be determined.

3. Results and discussion

3.1. Detection of ordered Pd–Y alloys

One of the effective criterions to determine the ordered degree for alloys is the decrement of electrical resistance. Fig. 1 shows the curve of R/R_0 vs. T. There is an inflection on the curve of as-quenched samples, but not for the as-annealed ones. From Ref. [5] we know that the electrical resistance anomaly variation is due to the order–disorder transition. The order–disorder transition temperature for Pd–7.52at.%Y alloy is 700±10°C.

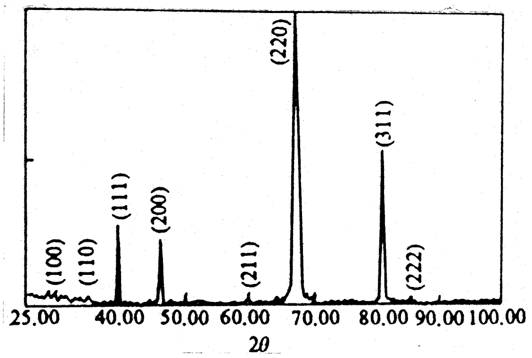

Fig. 2. XRD of annealed sample.

Fig. 2 shows the XRD pattern of the as-annealed sample. The Cu_3Au typed (that is Pd_3Y typed) LRO structure existed in the as-annealed alloy. Its lattice constant a_0 is equal to 3.9405×10^{-10} m. Compared with the matrix diffraction, the super-lattice diffraction is weak, sometimes it will not be displayed on the diagram, e.g. the (210) super-lattice diffraction peak does not exist in the figure. While other diffractions, such as (100), (110), (211), etc. are displayed in the diagram.

3.2. Microstructure

The microstructure for Pd–Y alloys is shown in Fig. 3. The average grain size for the as-quenched and as-annealed samples is 36.375 and 33.667 μm, respectively. After annealing, the grains distribute unequally and its size became smaller. For the quenched and annealed samples, the grain scale is almost the same, which is 750/mm^2 and 760/mm^2, respectively. Obvious twins existed in as-quenched samples, while for as-annealed ones twins decreased and sometimes even disappeared.

3.3. APB observation

Short range order (SRO) is known to occur in alloys composed of large and small atoms, where the neighbours of a small atom will be large ones more often than at random. Such a process is also encouraged by a large difference in electronegativity between the two types of atoms. Pd–Y alloys are ideal candidates for the appearance of SRO since there are very significant differences in both the atomic sizes and the electronegativities of the Pd and Y atoms. The appearance of SRO or partially ordered structure in the alloys can result in a relief of the strain energy characteristic of the disordered solid solution. When the composition of SRO alloy approach a certain atomic

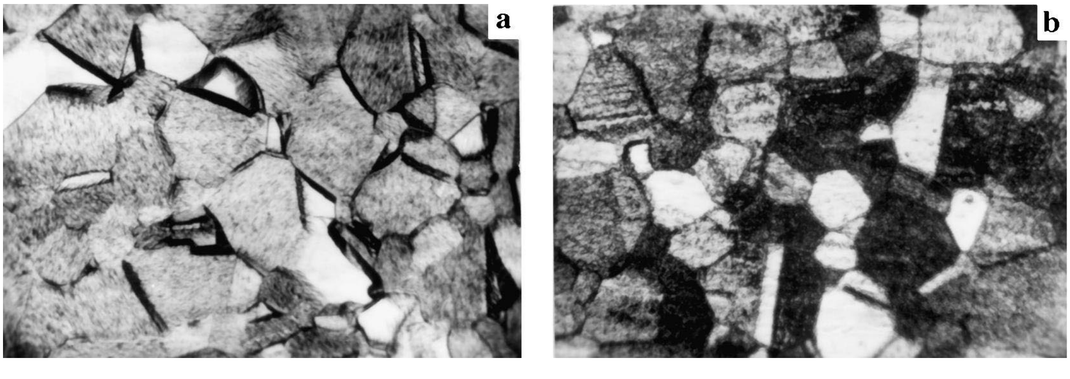

Fig. 3. Microstructure of Pd–8at.%Y alloys; (a) as-quenched; (b) as-annealed, magnification ×300.

percentage, being cooled slowly to a critical temperature from high temperature, the two kinds of atoms may arrange regularly in a wide region, and transfer to LRO structure. During the ordering process, a microregion with some atoms arranged orderly was formed in the alloys, which was called ordered domain, or anti-phase domain. The anti-phase domains arrange reversibly, and anti-phase domain boundaries (APB) exist. The temperature improves, the boundary moves, ordered domains gather and grow up until they contact each other, the LRO structure is formed. In order to stabilize the ordered structure, the energy should be decreased, attraction of different atoms must be higher than that of the same atoms.

APB may originate by two causes. First, it was the domain boundary formed during heat treatment processing. Second, it may be caused by dislocation. For some orientation, the energy of the domain boundary is low, these orientations may take superiority. The special feature of APB can be described by a lattice displacement across stacking fault. The lattice displacement vector of Pd_3Y (fcc) is $<1/2,1/2,0>$. A dislocation with Burgers vector goes through the ordered lattice, APB will be caused as it moves. A second dislocation across the same slip plane, and the stacking fault will be eliminated, which indicates that the Burgers vector of total dislocation in the disordered alloy is not equal to the vector of a lattice translation in the ordered super-lattice. The movement of these dislocations can cause APB. If the energy of the domain boundary is big enough, the dislocations with the same Burgers vector symbol slipped doubly are favorable for decreasing energy. Thus, in the simple typed superlattice, the second dislocation will eliminate the APB caused by the first one, and the APB zone was formed with these dislocations, and its geometry is similar to that of a couple of imperfect dislocations connecting with stacking faults. The energy equilibrium condition of APB relies on the interval between the two dislocations. The super dislocations are composed of these combinations and APB.

Using super-lattice diffraction, the contrast grade of APB may be caused, the image composed of light and dark stripes adjacently. The stripes parallel to the connection between domain boundary and membrane surface boundary. The extinction distance of superlattice diffraction is large, so the image of the domain boundary has only a few stripes. The APB of periodic anti-phase structure arranges regularly, APB stripes will also be continuous, and a loop formed around the domain boundary. In periodic anti-phase structure, the ordered domain arranges regularly, the image of the domain boundary arranges regularly, too. The cell-like structure of the as-annealed sample (shown in Fig. 4) is not complete, because in a certain extinction contour, not all APB has contrast grade. The solution was to lean the sample to let other super-lattice take effect and assure all APB should be disclosed.

In a word, Pd_3Y typed ordered phase existed in the as-annealed Pd–Y alloys, and anti-phase domain boundary was also found.

4. Conclusion

1. APB existed in the as-annealed Pd–Y alloys, it was composed of light and dark stripes adjacently. Incomplete cell-like structures were also found.
2. After annealing, Pd–8at.%Y alloy grains distribute unequally and its size becomes smaller, the grain scale for as-quenched and as-annealed samples was $750/mm^2$ and $760/mm^2$, respectively.

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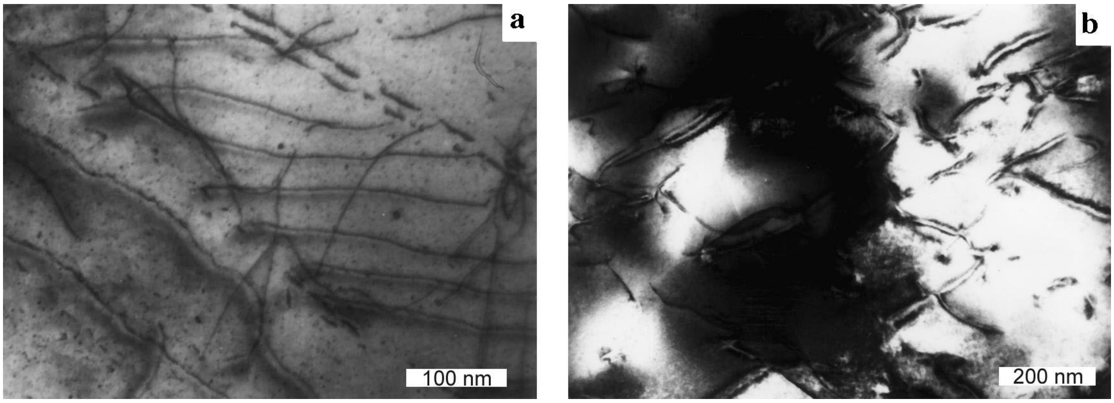

Fig. 4. TEM photos of as-annealed samples; (a) quench from 920°C; (b) quench from 940°C.