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A metastable BCC Si-substituted Pd solid solution

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Abstract. A metastable BCC Si-substituted Pd-rich $\text{Pd}_{100-x}\text{Si}_x$ solid solution Pd(Si) of the A2 type has been obtained in the concentration range $8 < x < 13$ by rapidly quenching the alloys from the liquid state. The solid solution decomposes into the equilibrium phases Pd and Pd_3Si depending on the composition and temperature. Once the Pd(Si) solid solution is formed, it remains stable up to 620 K before it decomposes under heat treatment.

1. Introduction

The Pd–Si system has been investigated several times regarding the crystal structures of different phases in this system [1–5], because $\text{Pd}_{100-x}\text{Si}_x$ alloys can be obtained in the amorphous state over a large concentration range by rapidly quenching them from the liquid state. During the course of our investigation of the crystallisation and melting behaviour of amorphous and crystalline $\text{Pd}_{100-x}\text{Si}_x$ alloys, we have observed that in the concentration range $8 < x < 13$ a new metastable Pd-rich Pd–Si solid solution forms, if the alloys in this concentration range are melt spun. In the present work, a preliminary study of the formation and crystal structure of this solid solution is presented.

2. Experimental details

$\text{Pd}_{100-x}\text{Si}_x$ ($0 < x < 13$) alloys, each of mass about 10 g, were prepared by high-frequency induction melting of the constituents in water-cooled quartz tubes in a purified argon gas atmosphere at a pressure of 400 mbar. During the very first melting, the Pd–Si reaction was found to take place extremely violently; therefore special care had to be taken to prepare the Pd–Si alloys. The purity of the starting elements was 99.999 wt% and 99.98 wt% for Si (supplier Ventron) and Pd (supplier Heraeus), respectively. The Pd–Si alloys were investigated in the as-cast state as well as in the amorphous state. The alloys in the amorphous state were obtained by melt spinning the alloys in a purified atmosphere of He gas at a pressure of 250 mbar on the surface of a rapidly rotating copper roller using quartz tubes with a melt ejection orifice of 0.5 mm [6]. The composition of the alloys was assumed to be that of the weighed components. The weight loss was found to be less than 0.01 wt%.

Room-temperature x-ray diffraction analysis (Mo $K\alpha$ radiation) was carried out, with an Enraf–Nonius Guinier camera. The crystallisation behaviour of the Pd–Si alloys

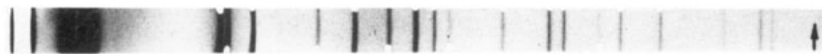


Figure 1. Room-temperature Guinier x-ray diffraction pattern of $\text{Pd}_{100-x}\text{Si}_x$ ($x = 12$), obtained by rapidly quenching from the melt at a roller speed of $3000 \text{ rev min}^{-1}$. The x-ray diffraction lines belonging to FCC Pd of the A1 type and BCC Pd(Si) of the A2 type are marked at the top and bottom, respectively. The arrow on the right is at $4\theta = 140^\circ$ from the primary beam mark on the left.

Table 1. Powder x-ray diffraction data of the Pd(Si) solid solution.

<i>h</i>	<i>k</i>	<i>l</i>	$\sin^2 \theta$		<i>I</i> (%)	
			Observed	Calculated	Observed	Calculated
0	1	1	0.0264	0.0266	100	100
0	0	2	0.0530	0.0532	52	18
1	1	2	0.0798	0.0798	78	37
0	2	2	0.1061	0.1064	32	11
0	1	3	0.1332	0.1330	48	16
2	2	2	—	0.1596	—	4
1	2	3	0.1862	0.1862	24	18

was investigated using the dynamic temperature x-ray diffraction (DTXD) method [7], in which the sample is heated or cooled at a constant rate, while the recording film is moving continuously across the diffraction slit. The heating rates used in this work were 100 K h^{-1} . The x-ray slit was 5 mm in width. The x-ray diffraction intensities were measured with a modified Zeiss-Jena (MD 100) microdensitometer.

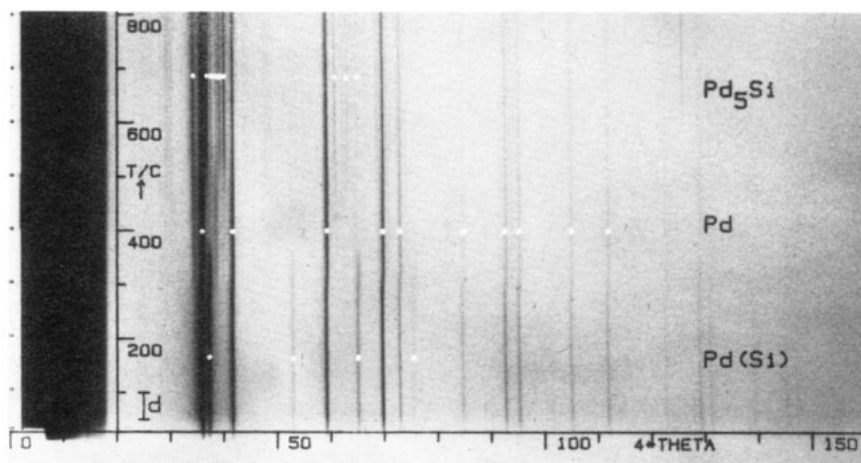


Figure 2. DTXD pattern of the crystalline $\text{Pd}_{100-x}\text{Si}_x$ ($x = 12$) alloy taken with $\text{Mo K}\alpha$ at a heating rate of 100 K h^{-1} . The x-ray diffraction lines labelled with white dots on a horizontal line are due to the phase printed on the right. *d* (at the left bottom) is the x-ray slit width.

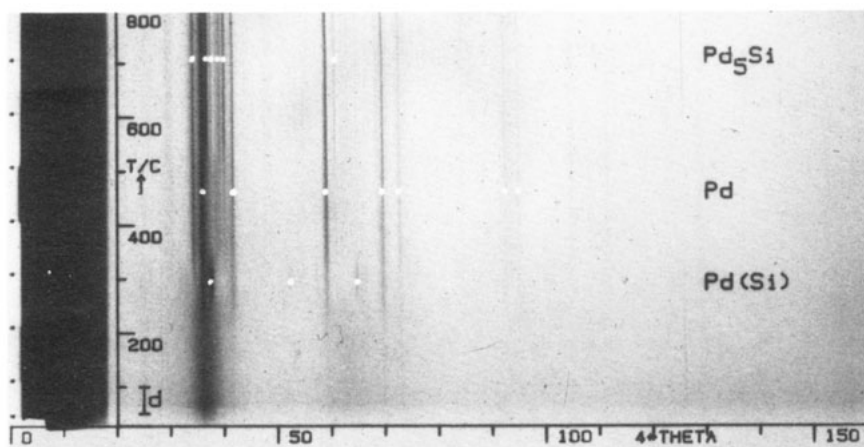


Figure 3. DTXD pattern of the amorphous $\text{Pd}_{100-x}\text{Si}_x$ ($x = 13$) alloy taken with Mo $K\alpha$ at a heating rate of 100 K h^{-1} . For meanings of symbols, see figure 2.

3. Results and discussion

With the melt-spinning roller speeds ($3000\text{--}5000 \text{ rev min}^{-1}$) of our apparatus, the $\text{Pd}_{100-x}\text{Si}_x$ alloys were found to be x-ray amorphous in the concentration range $13 < x < 28$. For $8 < x \leq 13$, however, the alloys were crystalline, if quenched at the rollerspeeds given above. From room-temperature x-ray diffraction analysis, two phases were identified in these crystalline alloys: a FCC Pd phase of the A1 type with $a = 3.889_8 \text{ \AA}$ and a BCC phase of the A2 type with $a = 3.081_2 \text{ \AA}$ (table 1 and figure 1). Whereas the former phase belongs to the equilibrium phase diagram as a terminal phase, the latter phase is a metastable Si-substituted Pd-rich Pd–Si solid solution (hereafter called Pd(Si)), because this solid solution has been found to form only if $\text{Pd}_{100-x}\text{Si}_x$ alloys for $x \leq 16$ were melt spun.

The DTXD analysis revealed that this solid solution decomposes and the equilibrium phases, Pd and Pd_5Si , at about 620 K ($= 347 \text{ }^\circ\text{C}$) are obtained (figure 2). If the amorphous $\text{Pd}_{100-x}\text{Si}_x$ ($13 < x \leq 16$) alloys were heated in the DTXD chamber, the first crystallisation product was found to be the BCC Pd(Si) solid solution at about 590 K ($317 \text{ }^\circ\text{C}$), before the equilibrium products, Pd and Pd_5Si , were observed at 620 K (figure 3).

The stability of the Pd(Si) solid solution, once formed, has been found to be high. At a heating rate of 100 K h^{-1} , the solid solution has been found to be stable up to about 620 K . Since this solid solution also forms after crystallisation of amorphous Pd–Si alloys (figure 3), before the equilibrium products are obtained, it is proposed that the amorphous Pd–Si alloys are distorted metastable solid solutions of the A2 type with crystallite size of the order of the A2-type elementary unit cell, if the crystallites have any meaning at such a small scale. Further investigations are in progress.

Acknowledgment

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