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Structural relationship between a titanium deuteride film and a thin protecting Pd toplayer.

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Thin films of titanium hydride (deuteride) have a number of possible applications as hydrogen (deuteride) storage material used in catalytic or energetic reactions (see [1] and Refs. therein). Application of such films under atmospheric pressure leads, however, to a partial decomposition of titanium hydride (deuteride) material due to interaction with oxygen [1]. In order to prevent interaction with air, titanium hydride (deuteride) films are covered by a protective layer such as Pd.

In this work titanium deuteride films (TiD_y) were obtained by the interaction of spectroscopically pure deuterium with thin Ti films evaporated in situ onto a Si(100) substrate within a glass UHV system [2], by evaporation of a fine Ti wire wound around a tungsten heater. During deposition of the films, the Si substrate was maintained at 273 K. The films were annealed for 60 min at 650 K. Deuterium was introduced in successive, calibrated doses at 298 K until an equilibrium pressure of 1 Pa was reached. Following this adsorption procedure, TiD_y films were routinely obtained with $y \approx 2$ [3]. The TiD_y films were then covered *in situ* by evaporation of a 10 – 12 nm thick Pd layer. The TEM specimens of the Pd/TiD_y films were prepared in cross-section (XS) according to the recipe described in Ref. [4], and TEM examination of the Pd/TiD_y films was performed *ex situ*. XS-TEM analysis was performed to obtain information regarding the bulk structure of corresponding films and to reveal the nanostructures formed at the Pd/TiD_y interface.

The results show the TiD_y film to be plastically deformed leading to an increase in the roughness of the top Pd layer (Fig. 1a). Selected Area Diffraction (SAD) analysis allowed the crystal phases in the bulk of the Pd/TiD_y film to be identified (Fig. 1b). By analysis of the cross-sectional TEM images the Pd/TiD_y interface was identified and structures which are the result of a crystallographic relationship between adjacent grains of the TiD_y top-layer, and between these grains and the Pd protecting layer could be characterized (Fig. 2). Complex structures, including Moiré patterns, have been identified (Fig. 2). The origin of these structures was determined by measuring the interplanar distances of the fringes in the lattice image [5], using well known formulas describing translational (d_T) and rotational (d_R) Moiré fringe spacing [6]. Selected Moiré structures, representing various lattice planes interfering within the Pd/TiD_y interface, are shown in Fig. 2 (spots A, B and C). Moiré fringes (A) (d_R = 1.48 nm) reveal the overlap of two TiD_2(111) lattices rotated through 10°. Moiré superstructure (B) (d_T = 1.73 nm) is produced by the overlap of TiD_2(111) and Pd(111). Moiré fringes (C) (d_T = 0.99 nm) disclose the cubic PdTi(111) transient phase [5], superimposed with the growth into the Pd(111) structure.

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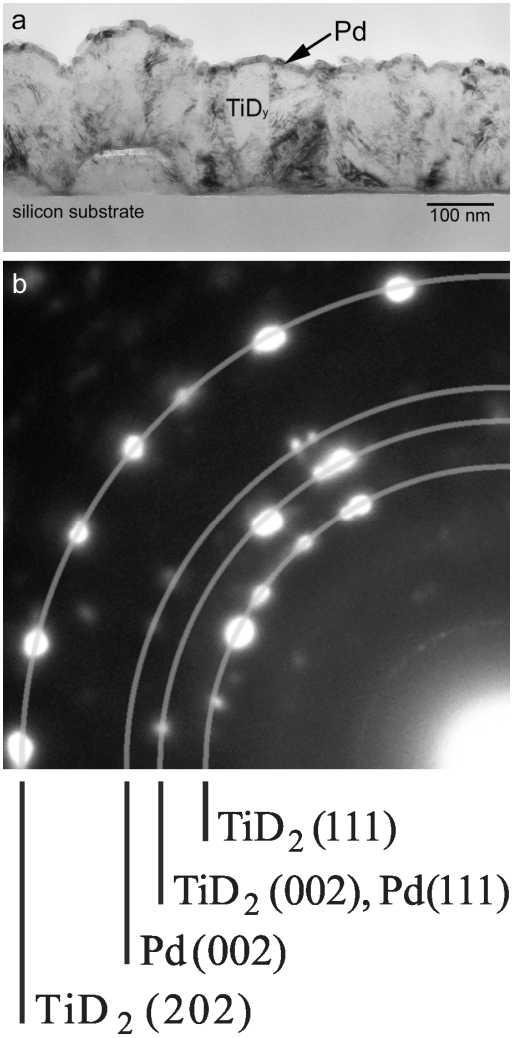

Figure 1. TEM cross-sectional Bright Field image of the titanium deuteride film covered by a 10 -12 nm thick Pd layer (a). The SAD pattern recorded on the Pd/TiDy area is shown in (b), together with the assignment of the most relevant low-index Miller planes of the diffraction spots.

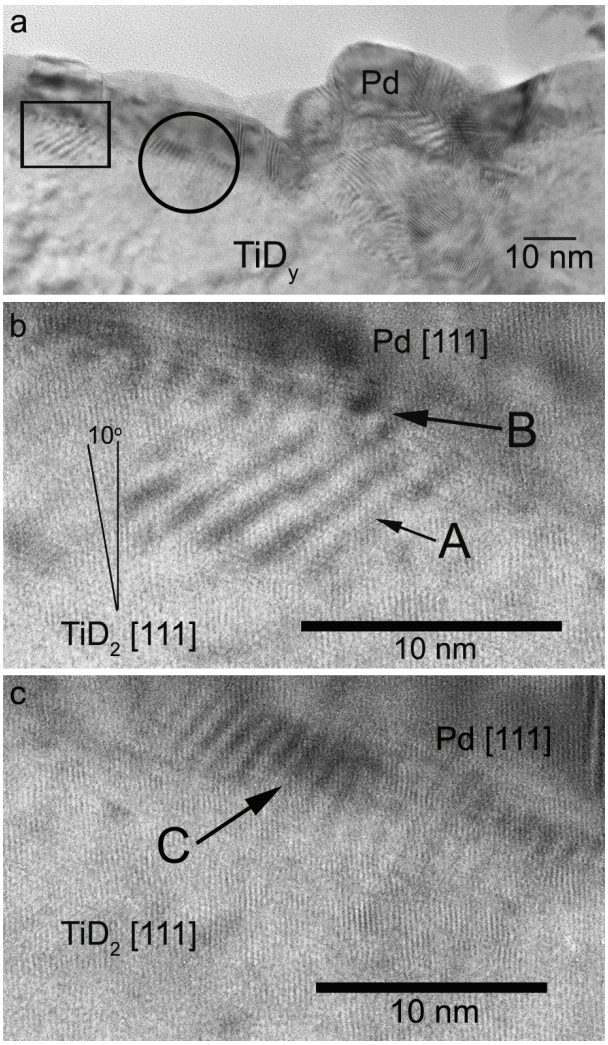

Figure 2. (a) Bright Field TEM image of the Pd/TiDy film in cross-section at high-magnification. Enlargements of the selected areas in (a): rectangular (b), circle (c). Spots A, B and C show selected Moiré structures (for a description see text).

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