

NOTE TO THE PAPER ENTITLED: TPD STUDY OF CARBON MONOXIDE INTERACTION WITH Pd/Nb BIMETALLIC SYSTEM

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New arrangement of temperature measurement by W-Ta thermocouple yields a slightly shifted temperature scale in comparison with that reported in the previous paper (Jirsák T., Nikolajenko V., Knor Z.: *Collect. Czech. Chem. Commun.* 59, 1709 (1994)). Temperatures resulting from the corrected thermoelectric voltages of the W-Ta thermocouple are given and figures with corrected temperature scales are presented.

Palladium multilayers deposited on the refractory metals (Nb, W, Ta and Mo) exhibit electronic properties of bulk Pd. Likewise, desorption temperature of carbon monoxide from these systems approaches that of bulk Pd itself. TPD data show that CO desorbs from Pd in range 450–500 K (refs^{1–3}). We found in ref.⁴ slightly lower desorption temperature of carbon monoxide (≈ 370 K) on thick Pd layer deposited on Nb. This discrepancy between our data and literature stimulated us to build a new arrangement of temperature measurement.

Thermocouples are currently used to measure the temperature of the studied sample in a wide range of temperatures. Several types of thermocouples can be used for temperatures up to 1 600 K. However, only two thermocouples are applicable above 2 100 K, namely: (i) WRe3–WRe25 and (ii) W-Ta. Calibration of the latter one is discussed in this paper.

The thermoelectric voltage of thermocouple was measured (see Fig. 1) in the range 295–900 K and compared with the literature values. After this procedure the measured thermal desorption temperature are slightly higher and thus agree with the published data. TPD data with corrected temperature scale are shown in Figs 2 and 3.

Precision calibration of a newly arranged W-Ta thermocouple yielded a detailed course of thermoelectric voltage of W-Ta thermocouple as a function of temperature which enabled accurate temperature measurements.

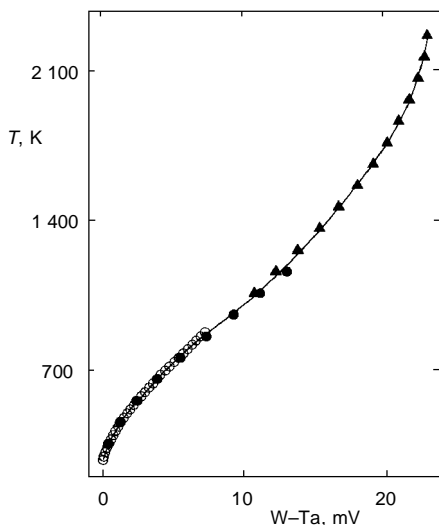


FIG. 1

Temperature (T) as a function of W - Ta thermoelectric voltage (W - Ta). \circ Our measurements, \bullet data from ref.⁵, \blacktriangle data from ref.⁶)

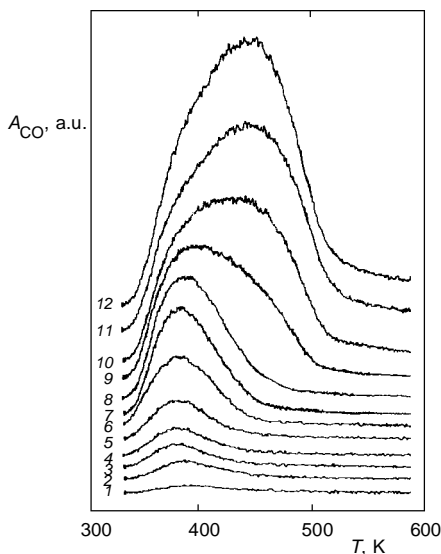


FIG. 2

CO TPD spectra (mass 28) as a function of Pd amount deposited on Nb. Pd deposited at 350–400 K, without annealing. CO adsorbed at 330 K (exposure 30 L). Time of Pd deposition: 1 20 s, 2 40 s, 3 1 min, 4 1 min 40 s, 5 2 min 20 s, 6 3 min 20 s, 7 5 min, 8 6 min 40 s, 9 8 min 20 s, 10 11 min 40 s, 11 16 min 40 s, 12 25 min

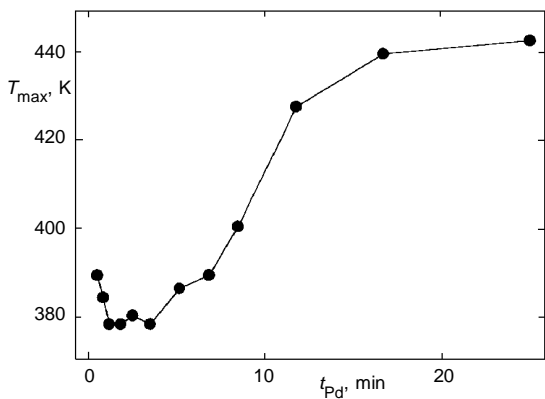


FIG. 3

Temperature maximum (T_{\max}) of CO TPD peak (mass 28) as a function of Pd amount deposited on Nb (expressed in deposition time, t_{Pd}). Pd deposited at 350–400 K, without annealing. CO adsorbed at 330 K (exposure 30 L)

EXPERIMENTAL

A new W-Ta thermocouple was spot-welded directly to the Nb sample (Nb ribbon 12.7×3.15 mm of thickness 0.115 mm). The length and diameter of Ta leads were arranged to minimize the temperature gradient along the sample. Thermoelectric voltage of our W-Ta thermocouple were checked ex situ at ambient pressure against a commercial thermocouple NiCr-CuNi (type E) exhibiting well defined thermoelectric voltages. The temperature 298 K was chosen as a reference point for W-Ta because it is more convenient for laboratory measurement than 273 K. Both thermocouples were mounted on metal element one close to each other in order to keep their temperatures at same value. Resistively heated ceramic furnace enabled to increase the temperature up to 900 K. The thermoelectric voltages of both thermocouples were recorded and the temperature was estimated from the known calibration curve of the NiCr-CuNi thermocouple.

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