



Investigation of the adsorption properties of borazine and characterisation of boron nitride on Rh(1 1 1) by electron spectroscopic methods



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ABSTRACT

The adsorption and dissociation of borazine were investigated on Rh(1 1 1) single crystal surface by Auger electron spectroscopy (AES), high resolution electron energy loss spectroscopy (HREELS) and temperature programmed desorption (TPD) methods. Borazine is one of the most frequently applied precursor molecules in the preparation process of boron nitride overlayer on metal single crystal surfaces. On Rh(1 1 1) surface it adsorbs molecularly at 140 K. We did not find any preferred orientation, although there is evidence of “flat” and perpendicular molecular geometry, too. Dehydrogenation starts even below 200 K and finishes until ~7–800 K. No other boron or nitrogen containing products were observed in TPD beyond molecular borazine. Through the hydrogen loss of molecules hexagonal boron nitride layer forms in the 600–1100 K temperature range as it was indicated by AES and the characteristic optical phonon HREEL losses of h-BN overlayer. The adsorption behaviour of the boron nitride covered surface was also studied through the adsorption of methanol at 140 K. HREELS and TPD measurements showed that methanol adsorbed molecularly and a fraction of it dissociated to form surface methoxy and gas phase hydrogen on the h-BN/Rh(1 1 1) surface.

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1. Introduction

The procedure of modification and self-assembly of nanostructures on surfaces is at present in the focus of surface science [1–10]. In this context, the epitaxial growth of ultra-thin layers of hexagonal boron nitride on transition metal surfaces has attracted a lot of attention. Inspired by the rich functionalities of graphene, scientists have a keen interest in the two-dimensional of hexagonal boron-nitride crystals (so-called “white” graphene). The h-BN possesses a number of advantageous properties over its carbon counterpart, including a constant wide band gap (5–6 eV) higher chemical inertness and thermal stability, enhanced oxidation resistance and good optical properties. These distinctions make h-BN uniquely attractive for applications in electronics, photonics, and nanocomposites [1]. Furthermore it is also a useful candidate in catalytic investigations as a well known chemically inert support material which does not interact electronically with the supported molecules or

metal clusters [2]. Particularly interesting is the h-BN/Rh(1 1 1) system, especially since the discovery of a self-organized boron nitride superstructure on a Rh(1 1 1) surface by Corso et al. [3]. It displays a h-BN nanomesh, into which molecules and nanoparticles can be readily adsorbed and thus arranged on the atomic length scale. It is also an interesting feature that a considerable structural difference does exist between h-BN overlayers formed on Pt(1 1 1) and Rh(1 1 1) despite the same symmetry and close lattice parameters [4]. On Pt(1 1 1) h-BN forms a rather flat single monolayer, while on Rh(1 1 1), it grows in a nanomesh form because of the stronger interfacial chemical bonding as a consequence of better orbital overlap.

There are two main ways to produce h-BN on the Rh surface. The first is the thermal decomposition of boron and nitrogen containing molecules (i.e.: borazine), however, its formation is also possible by the interaction of segregated boron [5] or dehydrogenation of decaborane [6] with N containing adsorbants, like NO. Following the latter route a clean single BN layer was prepared in monolayer or close to monolayer coverage through the interaction of NO with boron-containing polycrystalline Rh surface above 900 K [5]. This was proved by the appearance of intense Auger transitions

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at 176 eV (B) and 384 eV (N) above 400 K, which are typical for B–N species. The formation of boron nitride was also detected at 9.0–9.5 eV by UPS after this preparation. The observed photoemission can be attributed to the formation of σ bond between B and N. On the other hand in a wealth of preliminary works the preparation of h-BN was investigated on different substrates including e.g. Pd(1 1 1) [7], Cr(1 1 0) [8], Ni(1 1 1) [9] [1–10] and Refs. therein]. However, very few works deal with the chemistry of precursor molecules during the preparation steps of boron nitride overlayer [7,10,11]. Koel and co-workers investigated the adsorption of borazine on Au(1 1 1) and Pt(1 1 1) single crystal surfaces by electron spectroscopic methods [11], nevertheless to our best knowledge there is no similar work on Rh(1 1 1) surface until now.

Our main purpose was to reveal the adsorption properties of borazine on the Rh(1 1 1) single crystal surface by electron spectroscopic methods; furthermore we investigated the adsorption properties of the prepared boron nitride overlayer through the adsorption of methanol at low temperature. The surface stability of methanol on an inactive BN layer prepared on an active Rh(1 1 1) offers a possibility of comparison with data obtained on atomically clean Rh(1 1 1).

2. Experimental

The Rh(1 1 1) crystal used in this work was cut from a single crystal boule and was a product of the Material Research Corporation (purity 99.99%). It was mounted between two tantalum wires, which were connected via a copper block directly to a liquid nitrogen reservoir. Initially the sample was cleaned by repeated cycles of Ar⁺ sputtering (typically 1 kV, 1×10^{-7} mbar Ar, 300 K, 2 μ A for 10–30 min) and annealing to 900–1100 K until no contaminations were detected by AES and XPS. The base pressure in the chamber was around 1×10^{-9} Torr. The sample was heated resistively from 140 to 1200 K. Its temperature was monitored by a chromel–alumel thermocouple spot welded into the side of the crystal and was controlled with a feedback circuit to provide a linear heating rate of ca. 10 K/s. Gases were dosed through a 0.1 mm diameter capillary that terminated \sim 3 cm from the sample. The local pressure at the sample was about 10^{-7} mbar during dosing. The dosing temperature was \sim 140 K unless otherwise noted. The experimental work was performed in a two-level UHV chamber with a routine base pressure of 5×10^{-10} mbar produced by turbomolecular pump. The chamber was equipped with facilities for AES, XPS, HREELS and TPD. The HREEL spectrometer (LK, ELS 3000) is situated in the lower level of the chamber and has a resolution of 20–40 cm^{-1} (FWHM). The count rates in the elastic peak were typically in the range of 1×10^4 – 1×10^5 counts-per-second (cps). All spectra reported were recorded with a primary beam energy of 6.5 eV and at an incident angle of 60° with respect to the surface normal in the specular direction. Borazine of >99.8% purity was the product of Katchem Ltd. Borazine was stored at 254 K at all times except when charging the doser. All gas dosing lines were passivated and evacuated prior to borazine introduction.

3. Results and discussion

3.1. Preparation of the h-BN layer

3.1.1. Borazine adsorption on Rh(1 1 1) by AES and TPD

For the preparation of BN species the adsorption and decomposition of borazine on Rh(1 1 1) surface was investigated first by Auger-electron spectroscopy (Fig. 1). We applied two main preparation methods. We adsorbed borazine at low temperature (140 K) followed by annealing the sample to 900 K. The AES peaks characteristic of B–N species appeared on the spectra at 175 and 384 eV

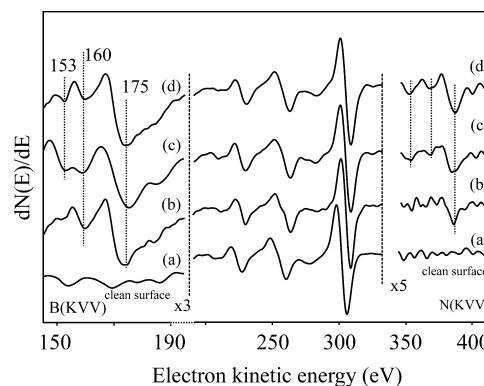


Fig. 1. AES spectra taken on the clean and borazine covered Rh(1 1 1) surface after \sim 24 L borazine adsorption at different temperatures. AES KVV lines of boron and nitrogen on clean surface (a) after borazine adsorption at 140 K (b), annealing the adsorbed layer to 900 K (c) and adsorption of borazine at 900 K (d).

kinetic energy [5]. When the adsorption of borazine was performed at higher temperature (900 K) the intensity of these peaks increased further. Dong and co-workers using a similar method (low dose adsorption at RT followed by high temperature annealing) concluded that the density of h-BN islands was higher than before when they prepared the surface layer only by high temperature adsorption [12]. In the new preparation process \sim 250 islands/ μm^2 appeared because of the 2D nucleation and growth. Auger electrons also carry information about the environment of their parent atoms. The energy separation of the multiplet lines is characteristic for the bonding partner and in this way this demonstrates some kind of fingerprint of the chemical environment of the excited atoms. Hence we also investigated the formation of the BN layer during annealing the surface after borazine adsorption at 140 K. The fine structure of the B (KVV) Auger series after borazine adsorption at 140 K and that after high temperature deposition differ in some distinctive features. In the latter case a three line Auger fine structure appeared on the spectra with characteristic energy separation marking the formation of a BN overlayer, which is differs significantly from the AES fine structure of B or B_2O_3 [13]. In contrast, when we adsorbed borazine at low temperature and investigated the condensed molecular layer we found just a very tiny third AES peak at 153 eV (see Fig 1a–d). Although the intensity and the resolution of the N (KVV) Auger lines are smaller with careful evaluation of the data we are able to observe the same enhancement of the characteristic Auger lines in the case of BN. This small difference in the fine structure of the AES spectra could be a sign of the completion of the dehydrogenation of borazine on the rhodium surface. Not only the fine structure of the B (KVV) AES lines but also the B(175)/Rh(306) peak to peak AES intensity ratio changed during annealing. After multilayer adsorption of borazine we reached \sim 0.20 B/Rh atomic ratio, which decreased immediately after a slight annealing caused by the low temperature desorption of condensed borazine layer (Fig. 2A). Above 300 K the ratio achieved a constant value at 0.12. With continuing exposure of the surface with increasing amount of borazine at 900 K we were able to attain a maximum B/Rh AES ratio of \sim 0.18–0.21. However, even raising the adsorption temperature to 1000 K did not result in a higher atomic ratio; instead it decreased slightly to \sim 0.165. The reason of this attenuation could be a mild alteration of the surface layer; STM results indicated that at this temperature (\sim 1000 K) the narrow, elongated h-BN islands transformed into more compact h-BN islands with a defective nanomesh superstructure [12]. We also checked the continuity of the BN overlayer by scanning the sample surface in consecutive Auger measurements and we didn't find any significant alteration of the AES B/Rh peak to peak intensity ratio (not shown)

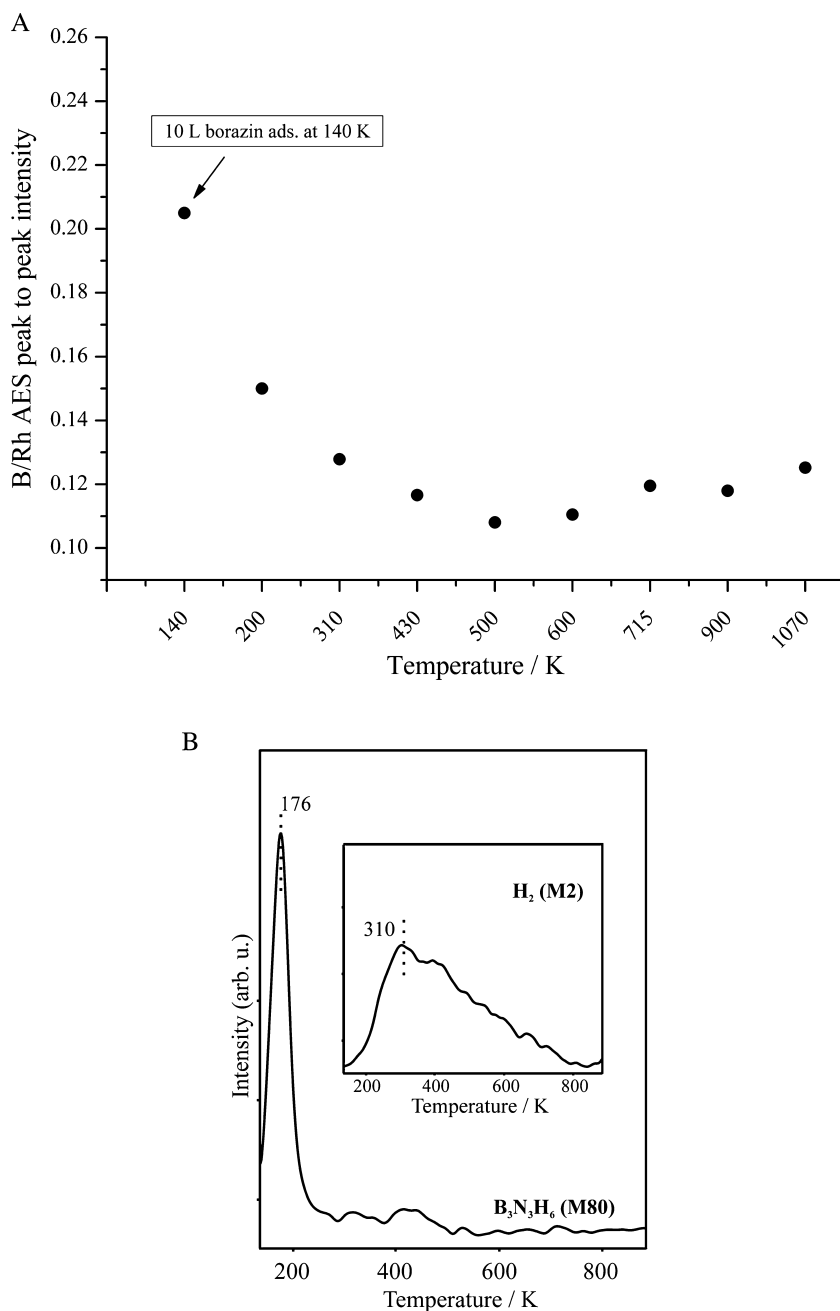
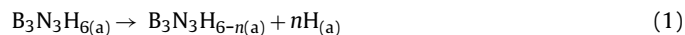


Fig. 2. Effect of annealing on the B(175)/Rh(306) peak to peak AES ratio after adsorption of borazine (~10L) on clean Rh(1 1 1) surface (A) and borazine (M80) and hydrogen (M2) TPD spectra (B) following 0.15L borazine adsorption on Rh(1 1 1) surface at 140 K.

which suggests a homogeneous dispersion of the BN overlayer on the Rh(1 1 1) surface at least as detected by AES.

Connecting to the AES results we studied the adsorption of borazine at lower exposures (0.15 L) at 140 K on Rh(1 1 1) and we followed the dissociation and desorption of possible reaction products of borazine with temperature programmed desorption techniques, TPD (Fig. 2B). In the temperature range of 100–1000 K the only desorption products were H₂ and B₃N₃H₆. TPD spectra for these two desorption products are displayed in Fig. 2B. Borazine desorbed from the surface with one sharp peak at $T_p = 176$ K, which is definitely connected to the desorption of the condensed adsorbate overlayer with zero order desorption kinetics (exposure dependence spectra not shown). Koel and co-workers found that on Pt(1 1 1) surface the exposure of 0.03 L borazine saturated the first chemisorbed layer and at larger doses a borazine multilayer

was formed [11]. In AES measurements we observed that 0.15 L exposure of borazine gives a ~0.12 B/Rh peak to peak atomic ratio at 300 K (at saturation) which is in good agreement with the value observed after annealing the condensed layer of borazine to RT. After multilayer desorption at ~200 K dehydrogenation reactions occurred. The supposed dehydrogenation reaction mechanism is the following:



TPD results proved that hydrogen desorption already started slightly below 200 K and continued – in a very broad temperature

Table 1
Characteristic vibrations of borazine on different single crystal surfaces.

Vibration mode	B ₃ N ₃ H ₆ gas-phase D _{3h} [11]	B ₃ N ₃ H ₆ on Pt(1 1 1) at 110 K [11]	B ₃ N ₃ H ₆ on Pt(1 1 1) at 170 K [11]	B ₃ N ₃ H ₆ on Au(1 1 1) at 110 K [11]	B ₃ N ₃ H ₆ on Au(1 1 1) at 180 K [11]	B ₃ N ₃ H ₆ on Rh(1 1 1) at 140 K [this work]	B ₃ N ₃ H ₆ on Rh(1 1 1) at 300 K [this work]
A ₂ ''	ν ₈ , γ-BH	918	915	910	910	920	920
	ν ₉ , γ-NH	719	710	710	710	720	730
	ν ₁₀ , γ-BN	394	400	400	400	410	405
E'	ν ₁₁ , ν _{as} -NH	3486	3485	3485	3460	3480	3480
	ν ₁₂ , ν _{as} -BH	2520	2535	2535	2490	2510	2530
	ν ₁₃ , ν _{as} -BN	1465	1465	1465	1460	1460	1450
	ν ₁₄ , ν _{as} -BN	1460					
	ν ₁₅ , δ-BH	1096					
	ν ₁₆ , δ-NH	990		955			
	ν ₁₇ , δ-BN	518		560(?)			

range – up to ~800 K (Fig. 2B). The TPD peak maximum at $m/e = 2$ (H₂) was observed around 310 K.

3.1.2. HREELS results

After a very high exposure of borazine (~45 L) on Rh(1 1 1) surface at 140 K we followed the effect of annealing on the HREEL spectra of the adsorbed layer (see Fig. 3A). At 140 K there were loss peaks at 410, 720, 920, 1050, 1460, 2510, 2980 and at 3480 cm⁻¹ which correspond well to the gas phase IR spectra of borazine [11]. The vibrational modes of borazine and their assignments are collected in Table 1. The peak at 1460 cm⁻¹ (E' representation/in-plane motions) belongs to the B–N asymmetric vibration, and the peaks observed with smaller intensity at 2510 and 3480 cm⁻¹ are connected with the E' B–H and N–H asymmetric modes, respectively. At 200 K all peaks decreased in intensity due to the multilayer desorption. Further heating of the adsorbed layer to 300 K led to the significant attenuation of the A₂'' modes at 410, 720 and 920 cm⁻¹ and at the same time the 1460 cm⁻¹ peak remained intense. Consequently the relative intensities of the peaks changed and at room temperature the latter peak dominated the spectra. At and above 500 K the spectra showed a completely different picture. The peaks from the B–H and N–H regions disappeared very likely due to the almost complete dehydrogenation reactions of borazine. Although the broadened peak at ~720–750 cm⁻¹ strengthened and above this temperature it was the most intense one on the spectra. We suppose that this peak belongs to a partially dehydrogenated fragment and it is not the reappearance of the γ–N–H peak, namely all the other peaks corresponding to the A₂'' representations are absent.

This idea is supported by the TPD measurements in which hydrogen desorption was observed in this temperature range (Fig. 2B). Although the loss at the N–H stretching region at 3480 cm⁻¹ was still detectable, it disappeared when we reached the temperature at which the dehydrogenation process completed (~700–800 K). Above 900 K the position of the peak at 720 cm⁻¹ shifted to higher wavenumbers (to 790 cm⁻¹). This peak belongs to the transverse optical (TO) phonon with out-of-plane polarization [14]. The remaining two main loss peaks are attributed to the phonons with in-plane polarization; the higher energy peak originates from the longitudinal optical (LO) phonon, and the lower energy one from the transverse optical phonon (see Table 2). The observed HREEL spectra at 1100 K correspond well to the literature data on hexagonal boron nitride layer (h-BN) [11]. Taking into account that in specular geometry only modes having a dynamic dipole component perpendicular to the substrate surface are allowed we can conclude in this case that due to a very strong contribution of the in-plane modes (E') a significant part of the molecules are oriented on the surface perpendicular or at least in slightly tilted position. This behaviour is similar to the results detected on Pt(1 1 1) surface, at the same time it is in contrast with

the results observed on Au(1 1 1) surface, where “flat” orientation geometry occurred [11].

To prove our presumption we followed the effect of exposure on the HREEL spectra after adsorption of borazine at 140 K (Fig. 3B). At low exposure (0.05 L) we observed only a little signal showing that borazine adsorbed on the surface and until 0.1 L the A₂'' modes dominated the spectra. This suggests that similarly to benzene on the Rh(1 1 1) surface [15] at low exposures and at low temperature borazine adsorbs in a planar geometry i.e. with the aromatic ring plane parallel to the metal surface. Nevertheless at higher coverage there is a mixed state of molecule geometries and we didn't perceive any strongly preferred orientation of the molecules with respect to the surface plane. The first case requires a more substrate specific chemical bonding mechanism between the molecules and the surface which is responsible for the perpendicular orientation. We suppose that (similarly to the previous results on Pt(1 1 1) surface [11]) a σ-type electron donation and interaction occurs from the ring N atom to the empty d states of rhodium. For such an interaction a dehydrogenated nitrogen atom is needed in the borazine molecules. Our TPD results proved that dehydrogenation reactions have already started below 200 K, in this way the explained mechanism could be possible on the Rh(1 1 1) surface.

3.2. Methanol adsorption on h-BN covered Rh(1 1 1)

The effects of a small amount of promoters including boron have been recognized a long time ago. Earlier we investigated the effects of segregated boron on the adsorption behaviour of various molecules (e.g.: CO₂, C₂N₂, O₂) on Rh surfaces [16–18]. Similarly to the case of adsorbed NO we also observed the formation of a stable boron nitride species from the interaction of CN_(a) with the segregated boron on the surface [17]. The adsorbed CO₂ and O₂ on boron containing Rh surfaces showed high affinity for the formation of stable boron oxide [16,18].

In the present work we investigate the adsorption properties and the reactivity of a relatively inert h-BN layer on Rh(1 1 1) surface. Methanol adsorption was chosen to test the catalytic properties of the h-BN layer and in this way we also used methanol to characterize the prepared BN layer. We adsorbed ~6 L of methanol

Table 2
Characteristic vibrations of BN species on different single crystal surfaces.

System	^a TO _⊥ (cm ⁻¹)	^a TO (cm ⁻¹)	LO (cm ⁻¹)
h-BN/Ni(111) [22]	728	1360	1360
h-BN/Pd(111) [22]	784	1384	1432
h-BN/Pt(111) [22]	792	1384	1464
h-BN/Rh(111) [this work]	790	1360	1460, 1510
Bulk h-BN [22]	776, 824	1352, 1360	1600

^a TO_⊥ transverse optical phonons with out-of-plane, LO (longitudinal optical) and TO_{||} phonons with the in plane polarization.

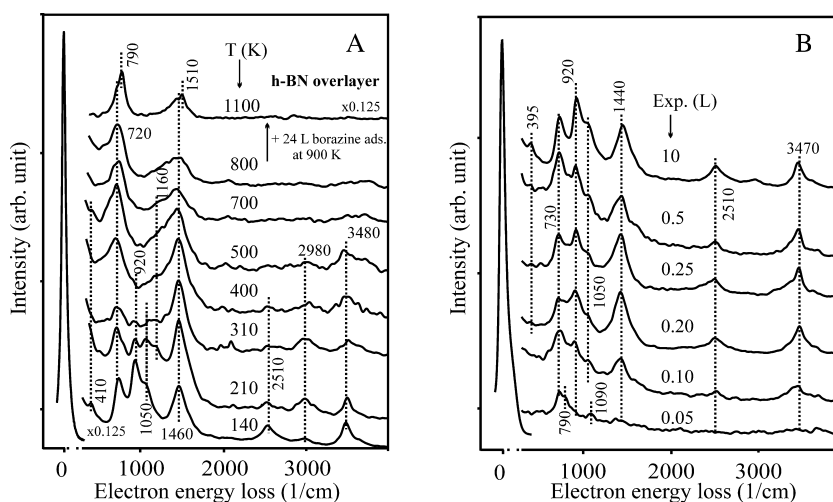


Figure 3. (A) Effect of annealing on the HREEL spectra after ~ 10 L borazine adsorption on clean Rh(111) surface and (B) effect of exposure after adsorption of borazine at 140 K.

on the clean and BN covered surface at 140 K and followed the effect of annealing on the HREEL spectra (Fig. 4A and B). The appearance of the loss features from the various vibration modes of methanol on the surface layer at 710, 1090, 1140, 1465, 2980 and 3260 cm^{-1} suggests that methanol adsorbs molecularly on the clean and also on the h-BN covered Rh(111) at 140 K. The appearance of these losses agrees well with the gas phase vibrational data of methanol [19]. An interesting feature is that the two main losses corresponding to the BN layer are also observable on the spectra taken at 140 K (Fig. 4B), however, the intensity ratio of the two peaks changed at this temperature. The main difference between the HREELS results observed on the clean and BN covered layer that on the clean surface we observe a significant loss at 400 K due to the C–O stretching vibration belongs to the decomposition product of methanol bonded to a surface rhodium atom.

As the TPD results showed the weakly bonded molecules desorbed from the surface at ~ 160 K (Fig. 4C). This caused a significant attenuation of the loss peaks of methanol below 200 K in the HREEL spectra, but it is also clear that a small part of the molecules remained on the surface. These molecules either stayed in molecular form or decomposed—probably to methoxy and hydrogen suggested by the disappearance of the $\delta(\text{OH})$ vibration loss at 3350 cm^{-1} and the presence of the features at 710, 1085, 1140, 1465

and 2940 cm^{-1} belonging to methoxy species [20]. In a former study on clean Rh(111) we got similar results, namely that methanol that was adsorbed at 100 K on the clean surface desorbed below 300 K in four different peaks at ~ 135 , 148, 200 and 255 K as a sign of multilayer and recombinative desorption processes. The most stable part of the molecules decomposed at higher temperature and provided H_2 and CO as desorption products at 360 and 490 K [21]. On BN covered Rh(111) we observed the above mentioned narrow losses (connecting to methoxy species) up to 400 K (Fig. 4B), which suggested a considerable stability of the methoxy species on this inert surface. TPD spectra also corroborated that methanol desorption completed via recombinative desorption only around 400 K. Although we pointed out the presence of a small fraction of H_2 ($T_p \sim 350$ K) and CO ($T_p \sim 490$ K) in the TPD spectra close to the detection limit of our MS (not shown) – in contrast to the clean surface – no signals of carbon-monoxide were observed in the HREEL measurements on the boron nitride covered Rh(111) surface. In the light of our previous results observed on clean surface, the lack of CO losses in the HREELS suggests that the Rh(111) surface fully covered by boron nitride layer. Above 400 K all of the losses belonging to hydrocarbon species disappeared from the HREEL spectra and only the vibrations characteristic of the h-BN overlayer were observable.

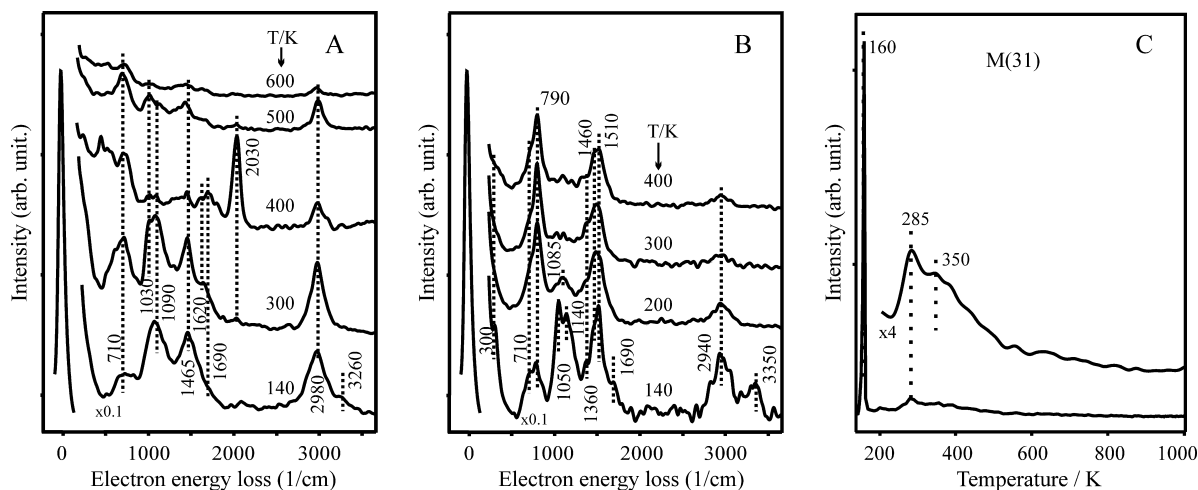


Figure 4. Effect of annealing on the HREEL spectra after ~ 6 L methanol adsorption on the clean (A) and h-BN covered (B) Rh(111) at 140 K. TPD spectra of methanol (M31) on h-BN covered Rh(111) at 140 K (C).

4. Conclusions

- We provided fingerprint data by Auger spectroscopy that makes it possible to differentiate between adsorbed borazine multilayer and h-BN overlayer.
- Borazine adsorbed molecularly at 140 K on the clean Rh(1 1 1) surface, and dehydrogenation reactions started even below 200 K. Hydrogen desorption took place in a wide temperature range from 190 K to 800 K. No other boron or nitrogen containing products were observed in the TPD spectra.
- Boron nitride layer formation began above 600 K as indicated by the AES and HREELS measurements. The strong characteristic losses of well defined h-BN appeared at ~1000 K in the HREEL spectra.
- Borazine adsorbs in a “flat” position on the Rh(1 1 1) surface at low exposures but at higher coverage perpendicular or slightly tilted positions dominated the geometry of adsorbed molecules. Nevertheless at multilayer coverage we didn't perceive any strongly preferred orientation of the molecules with respect to the surface plane.
- Methanol adsorbed molecularly on BN covered Rh(1 1 1) at 140 K. A small part of the molecules were stable up to 400 K on the surface. We didn't find any sign of decomposition products in contrast with the clean surface where CO produced and desorbed above 400 K.

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