Study of UO2 with ice on surface

**PK: preparation chamber**

**AK: analysis chamber**

**ov: overview**

**RT: room temperature**

**RGA: Residual gas analyser**

**08/02/2012**

Machine opened, water inlet valve and tubes installed

Machine baked out.

**09/02/2012**

Gold sample introduced and Electron Energy Analyser calibrated.

Si-substrate introduced and kept in 2nd pump stage (about 10-7 mbar base vacuum) for 15min. At 300°C

**Initial films deposition (to clean the U source by sputtering it)**

**01/03/2012**

 **Time fil. work. target1 pressure Ar pressure O2**

Ov.001 120sec. 4.4V/3.7A 38V/43mA 700V/3mA 1.0e-6mbar -

U4f.002

O1s.003

Ov.004 180sec. 4.4V/3.7A 42V/ 40mA 700V/2.7mA 6.0e-7mbar -

U4f.005

O1s.006

C1s.007

Ov.008 180sec. 4.4V/3.7A 40V/39mA 700V/ 2.7mA 9.0e-7mbar 8e-5torr

U4f.009

O1s.010

Ov.011 180sec. 4.4V/3.8A 40V/43mA 700V/ 3.1-2.8mA 8.0e-7mbar 1e-4torr

U4f.012

O1s.013

**02/03/2012**

HeII.014

Sample ejected

New Si-substrate introduced 2nd pumping stage for 10min. heated at 200°C (substrate outgassing).

**Oxide films prepared by reactive sputter deposition (in presence of O2 partial pressure)**

Ov.015 300sec. 4.5V/3.8A 40V/45mA 700V/ 3.5-3.3mA 7.4e-7mbar 2e-4torr

U4f.016

C1s.017

HeII.018

HeI.019

Ov.020 180sec. 4.4V/3.7A 44V/36mA 700V/ 3.3-3mA 7.5e-7mbar 1e-3torr

U4f.021

C1s.022

HeII.023

HeI.024

Cool down to N2(l) Temp (-6 mV type K thermocouple) measured -5.5 mV (~ - 185 °C with 25 °C = 0.1 mV), liquid nitrogen running through rod.

**Below Temperature is indicated by the Thermocouple voltage (consult conversion table)**

Add water into PK 8e- torr 180 s (~ L).

HeII.xxx Unfortunately, no measurement was possible, the signal was constantly around zero. PC restarted

HeII.025 temp at -3.3mV, ice was presumably present because when I rode in AK and the temp was at -5.5mV, RGA showed mass 18:1e-9mbar

 Well at temp. -3.3mV was RGA Mass 18: 2e-7mbar

Lunch break UV source off!!

HeII.026 Temp. -1.0mV well, that was probably nothing

HeII.027 Temp. -0.9mV

Ov.028

Uf4.029

**05/03/2012**

Leave HeII.030 film about weekend in AK.

Water tank checked and pumped out again.

Fresh UO2 film (deposited on the previous one)

 **Time fil. work. target1 pressure Ar pressure O2**

HeII.031 180sec. 4.4V/3.8A 42V/503mA 700V/ 3.6-3.2mA 8.3e-7mbar 1e-4torr

Ov.032

U4f.033

Cool down to N2(l) Temp (-6 mV ?) measured -5.6V (~ - 1 °C with 25 °C = 0.1 mV), N(l) running through rod

Add water into PK 1e-5torr, 180s (~L)

Hell.034 RGA: Image1

HeII.035 +5min. RGA: Image2

HeII.036 + 10min. RGA: Image 3 Temp: -4.5mA

HeII.037 +12min. RGA: Image4 Temp:-4.3mA AK:1.2e-7mbar

HeII.038 +17min. RGA: Image5 Temp:-4.1mA AK:8.0e-7mbar

Strong increase in pressure: water AK:4e-6mbar RGA:image6

HeII.039 + 21min. RGA: Image7 Temp: -3.7mA AK: 6.8e-6mbar

 RGA: Image8 Temp:-3.6mA AK: 1.1e-5mbar

HeII.040 +24min. RGA: Image9 Temp: -3.4mA AK: 7.1e-6mbar

HeII.041 +28min. RGA: Image10 Temp: -3.2mA AK: 1.5e-6mbar

HeII.042 +32 mins. RGA: Figure11 Temp: -3.0mA AK: 8.2e-7mbar

HeII.043 +39 mins. RGA: Image12 Temp: -2.6mA AK: 4.6e-7mbar

HeII.044 +2h 5min.

**06/03/2012**

**UO3 film prepared by O in new machine and deposition/desorption of an ice layer**

045 ov

046 U4f

047 O1s

048 HeII

049 HeI

Cool down to N2(l) Temp (-6 mV ?) measured -5.6V (~ - 1 °C with 25 °C = 0.1 mV), N(l) running through rod

Add water into PK 8e-6torr, 180s (~L)

050 HeII 0min. RGA: Fig.13 Temp.-5.7 AK: 2.3e-8mbar

total ice surface

051 HeII +5min. RGA: Image 14 Temp.-5.1 AK: 3.7e-8mbar

052 HeII +8 min. RGA: Image 15 Temp.-3.7 AK: 2.7e-6mbar

053 HeII +11 min. RGA: Image 16 Temp.-3.2 AK: 2.1e-5mbar

054 HeII +15min. RGA: Image 17 Temp.-3.1 AK: 7.4e-7mbar

055 HeII +20 min. RGA: Image 17 Temp.-2.8 AK: 6.1e-7mbar

 Heating with compressed air

056 HeII +2 3min. RGA: Image18 Temp.-2.3 AK: 4.5e-7mbar

057 HeII +2 6min. RGA: Image 19 Temp.-1.7 AK: 3.8e-7mbar

058 HeII +30 min. Temp.-1.5 AK: 3.3e-7mbar

059 HeII +33min. Temp.-1.2 AK: 2.6 e-7mbar

 Rod moved in 200 steps further to measure next to the UV spot!

060 HeII +36min. Temp.-1.1 AK: 2.4E-7mbar

**07/03/2012**

061 HeII film from yesterday measured again. NO change recognizable!

**New UO3 film deposition**

062 ov

063 u4f

064 O1s

065 HeII

066 HeI

Cool down to N2(l) Temp (-6 mV ?) measured -5.4V (~ - 1 °C with 25 °C = 0.1 mV), N(l) running through rod

Add water into PK 8e-6torr, 180s (~L)

067 HeII 0min. RGA: Image20 Temp.-5.6 AK: 2.8e-8mbar

068 HeII +3min. RGA: Image21 Temp.-4.5 AK: 4.4e-8mbar

069 HeII +6min. RGA: Image22 Temp.-3.6 AK: 3.0e-6mbar

070 HeII +9min. RGA: Image23 Temp.-3.3 AK: 4.5e-6mbar

071 HeII +12min. RGA: Image24 Temp.-3.1 AK: 8.0e-7mbar

072 HeII +15min. RGA: Image25 Temp.-2.9 AK: 5.0e-7mbar

073 HeII +18min. RGA: Image26 Temp.-2.8 AK: 4.0e-7mbar

From now on, the rod is heated with compressed air

074 HeII +21min. RGA: Image27 Temp.-1.7 AK: 4.0e-7mbar

075 HeII +24min. Temp.-1.2 AK: 3.1e-7mbar

076 HeII +27min. Temp.-0.9 AK: 2.7e-7mbar

Try driving 200 steps further in!!

077 HeII +32min. Temp.-0.7 AK: 2.2e- 7mbar

078 HeI

Lunch break!

The same sample is again covered with ice and this time "thawed" more slowly

Cool down to N2(l) Temp (-6 mV ?) measured -5.4V (~ - 1 °C with 25 °C = 0.1 mV), N(l) running through rod

Add water into PK 8e-6torr, 180s (~L)

079 HeII 0min. RGA: Image28 Temp.-5.6 AK: 4.1e-8mbar

080 HeII 5min. Temp.-5.6 AK: 3.7e-8mbar

081 HeII 10min. Temp.-5.6 AK: 3.4e-8mbar

082 HeII 15min. RGA: Image29 Temp.-5.6 AK: 3.4e-8mbar

Start with the slow warm-up N2 pressure throttled to 0.5 bar!

083 HeII 20min. Temp.-5.6 AK: 5.0e-8mbar

084 HeII 25min. RGA: Image30 Temp.-4.8 AK: 5.6e-8mbar

085 HeII 29min. RGA: Image31 Temp.-4.0 AK: 1.0 e-6mbar

086 HeII 32min. RGA: Image32 Temp.-3.8 AK: 3.9e-6mbar

087 HeII 35min. RGA: Image33 Temp.-3.8 AK: 2.8e-6mbar

088 HeII 38min. Temp.-3.4 AK: 2.8e-6mbar

089 HeII 42min. RGA: Image34 Temp.-2.9 AK: 1.3e-6mbar

090 HeII 45min. RGA: Image35 Temp.-2.3 AK: 7.5e-7mbar

091 HeII 50min. Temp.-2.0 AK: 6.0e-7mbar

092 HeII 55min. Temp.-1.7 AK: 4.8e- 7mbar

093 HeII 60min. Temp.-1.5 AK: 3.8e-7mbar

Rod drove 400 steps farther in...

094 HeII 63min. Temp.-1.5 AK: 3.5e-7mbar

Rod returned to original position (-400)

095 HeII 66min. Temp.-1.5 AK: 3.5e-7mbar

096 HeII 70min. UV source was off for a moment, restarted, test if the result is the same as before.

097 HeI

**08/03/2012**

098 ov Sample left in AK overnight. Pressure: 1.8e-8mbar

099 uf4

100 O1s

101 HeII

102 HeI

Cool down to N2(l) Temp (-6 mV ?) measured -5. 4V (~ - 1 °C with 25 °C = 0.1 mV), N(l) running through rod

Add water into PK 8e-6torr, 180s (~L)

103 HeII 0 min. RGA: Image36 Temp.-5.6 AK: 2.7e-8mbar

104 HeII +5 min. Temp.-5.6 AK: 2.1e-8mbar

Start with the slow warm-up N2 pressure throttled to 0.65 bar!

105 HeII +10 min. Temp.-5.7 AK: 1.9e-8mbar

Heater switched on to warm up slowly: 3V/0.4A = 1.2W

106 HeII +16 min. Temp.-5.7 AK: 1.8e-8mbar

N2 pressure reduced to 0.65 bar

Heater: 5V/0.6A = 3W

107 HeII +20 min. RGA: Fig.37 Temp.-5.6 AK: 1.8e-8mbar

Heater: 8V/1.0A = 8W

108 HeII +24 min. RGA: Image38 Temp.-5.5 AK: 2.5e-8mbar NITROGEN has outgassed!!

109 HeII +2 8min. RGA: Image39 Temp.-5.3 AK: 1.8e-8mbar

Heater: 9V/1.2A = 10.8W

110 HeII +32 min. RGA: Image40 Temp.-5.1 AK: 1.9e-8mbar

Heater: 10V/1.3A = 13W

111 HeII +36 min. RGA: Fig.41 Temp.-4.5 AK: 3.5e-8mbar

Heater: 9.4V/1.2A = 11.3W

112 HeII +38 min. RGA: Image42 Temp.-4.3 AK: 8.3e-8mbar

113 HeII +41 min. RGA: Image43 Temp.-4.1 AK: 5.2e-7mbar

During the measurement 113 heating reduced to 6V/0.8A = W. As a result, the pressure of 6.6e-7 immediately decreased to 1e-7.

114 HeII +41 min. RGA: Image44 Temp.-4.6 AK: 6.2e-8mbar

It's getting colder again...

heating back to 9.4V/1.2A = 11.3W,

115 HeII +50 min. RGA: Image45 Temp.-5.0 AK: 4.0e-8mbar

it's getting colder..

Heater: 10V/1.3A = 13W

116 HeII +54 min. Temp.-4.9 AK: 3.4e-8mbar

117 HeII +57 min. Temp.-4.6 AK: 3.4e-8mbar

118 HeII +60 min. Temp.-4.5 AK: 3.8e-8mbar

Heating: 10.4V/1.3A = 13.5W aahhh here we go! I'm back to 10V, pretty sluggish regulation...

119 HeII +6 4min. RGA: Image46 Temp.-4.1 AK: 3.1e-7mbar

120 HeII +68 min. RGA: Image47 Temp.-4.0 AK: 8.9e-7mbar

121 HeII +72 min. RGA: Image48 Temp.-3.7 AK: 4.3e-6mbar

122 HeII +75 min. RGA: Image49 Temp.-3.5 AK: 4.3e-6mbar pressure drops again, water is gone!

123 HeII +78 min. RGA: image50 Temp.-3.6 AK: 8.3e-7mbar

124 HeII +82 min. Temp.-3.4 AK: 6.2e-7mbar

125 HeII +85 min. Temp.-3.3 AK: 5.2e-7mbar

126 HeI

127 ov +240min. AK: 6.3e-8mbar

128 u4f

129 o1s

**09/03/2012**

130 HeII sample from yesterday measured again

131 HeII rod moved to Pos:-800, sample should not be reduced here.

132 HeII rod moved to Pos:+-0, sample is not reduced here. Start 10:15AM

Comparison with original sample (HeII.065). Is there a very, very slight reduction?

TRIAL: Expose the sample to UV light only and see if there is a reduction.

133 HeII after 15 min.

 Was outside for a short time, UV source was off, currently not running very stable! (~10 minutes?!)

 Now let them work at higher pressure: 23A/72V gas valve: 5.0

134 HeII +15min.

135 HeII +30min.

136 HeII +45min.Source went out again. crap thing!

137 HeII +60min.

138 HeII +75min.

139 HeII +90min.

lunch break

140 HeII same sample, UV source was off. +4h45min.

141 HeII Rod: +200

142 HeII Rod:-1000

143 HeII Rod:-600

144 HeII Rod: xxx???

Weekend!

**Attention: He line is pumped out over the WE**