

28 **1. Introduction**

29 Nanoscale X-ray sensors are key elements to achieve even higher spatial
30 resolution in different X-ray based experiments, including imaging, diffraction, and
31 spectroscopy. A decrease of the sensor size down to nanoscale is required for further
32 advances in materials science, diagnostic and therapeutic healthcare, industrial process
33 control and homeland security since many X-ray techniques are key research methods
34 in these fields [1–3]. Nowadays, X-rays can already be focused down to sizes below 10
35 nm [4–6], while the conventional micron-scale direct-direction sensors lack the
36 necessary resolution [7]. Typically, X-ray detection can be realized using indirect and
37 direct methods, depending on how the radiation is transformed into a usable signal [8].
38 Indirect detection systems involve two steps which include X-ray conversion to visible
39 light with a scintillator material and subsequent light detection via a pixelated sensor
40 with photodiodes, in which the resolution is limited by the diffraction limit [9]. Direct
41 detectors convert X-rays to an electrical signal directly that results in a significantly
42 higher electrical signal per absorbed X-ray photon than in the previous case and are
43 considered more preferable in a majority of hard X-ray detection applications [8]. There
44 is still a demand for broadband, high sensitivity, and low-cost X-ray detectors, which
45 operate at room temperature [1].

46 Conventional direct-detection semiconductor sensors are usually fabricated
47 from bulk or thin-film materials, which makes further miniaturization and energy
48 consumption reduction more sophisticated [10]. Amorphous Se is commonly used in
49 mammography for low-energy X-ray beam detection, p-type Si finds applications for
50 dose measurement and beam imaging, while high atomic number Z materials, such as
51 Ge, GaAs, CdTe, and CdZnTe, are used at moderate and high X-ray energies, e.g., for
52 security screening [1,8]. The working principle of such sensors is based on a conversion

53 of the absorbed X-ray photon energy to a readable electronic signal. An X-ray photon,
54 when absorbed in the matter, creates a primary high-energy electron via the
55 photoelectric effect, which subsequently gives its energy to the surrounding lattice to
56 create an avalanche of secondary electron-hole pairs. Therefore, one absorbed X-ray
57 photon can generate a large number of charge carriers that can be detected
58 electronically. However, as the X-ray absorption is proportional to the volume of the
59 material, conventional semiconductor X-ray detectors still have pixel dimensions in the
60 order of tens of micrometers to obtain usable signal [7]. On the other hand, in devices
61 based on nanostructured materials, which have smaller volume but very high crystalline
62 quality and large surface-to-volume ratio, significantly more generated charge carriers
63 per absorbed X-ray photon can be extracted than in bulk detectors [11].

64 During the last two decades one-dimensional (1D) semiconductor
65 nanostructures, such as nanowires (NWs), have been extensively investigated for
66 various applications in nanoelectronic and optoelectronic devices, such as transistors,
67 photodetectors, and LEDs [12–14], because of their promising electrical and optical
68 properties, which arise from the high surface-to-volume ratio and size confinement
69 effects. The detection of light in the visible, infrared, and ultraviolet ranges using
70 individual NW devices has been a particularly active research topic, mainly due to the
71 high responsivity and fast response speed of single-crystal semiconductor NWs [15–
72 18]. However, there are very few reports on X-ray beam induced current (XBIC)
73 measurements in 1D semiconductor nanostructures. Ronning *et al.* have studied
74 Ga/GaAs heterojunction embedded in a silicon NW with a SiO₂ shell in detail using a
75 nanofocused X-ray beam and a combinatorial approach [19] as well as demonstrated
76 GaAs NW hard X-ray detection capability [20]. Wallentin *et al.* have extensively
77 employed synchrotron radiation and the XBIC method together with X-ray

78 fluorescence and diffraction measurements in InP and InGaP NWs to study doping [21],
79 mechanical strain [22], charge carrier collection in single-NW solar cells [23], electrical
80 detection of X-ray absorption fine structure [24] and to develop a novel nanoscale
81 measurement technique for a beamline [25]. The same group of collaborators has also
82 demonstrated X-ray detection and beam shape characterization using individual NWs
83 either perpendicular [11] or parallel [26] to the X-ray beam propagation direction.
84 Furthermore, Li *et al.* have demonstrated X-ray detection using individual ZnCdTe
85 nanoribbons [27], while other studies only report the use of networked 1D
86 nanostructures [28,29]. Thus, for the development of novel efficient high-resolution X-
87 ray detectors based on NWs in the future, the detection of X-rays using NWs from
88 various semiconductor materials containing elements with different Z-numbers
89 deserves a more thorough study.

90 This study demonstrates the detection of X-ray radiation using individual CdS,
91 SnO₂, and ZnO NWs. Electrical measurements were performed under ambient
92 conditions while exposing two-terminal individual NW-based devices to soft X-rays
93 with continuous spectrum generated by a conventional X-ray tube with a tungsten
94 anode. The high nanoampere-range XBIC measured in the NW devices could mainly
95 be attributed to the efficient transport and collection of generated charge carriers due to
96 the single-crystalline nature of NWs and the short NW length. Such fast-response and
97 high-sensitivity nanoscale X-ray detectors might be useful for sub-micron resolution
98 imaging and nanofocused beam shape measurements.

99

100 **2. Experimental section**

101 *2.1. Nanowire synthesis and characterization*

102 CdS, SnO₂ and ZnO NWs were synthesized via atmospheric pressure chemical
103 vapor transport method in a horizontal quartz tube reactor. The NWs were grown on
104 oxidized silicon wafers SiO₂/Si(100) (*Semiconductor Wafer, Inc.*) coated with spherical
105 Au nanoparticles (NPs, *Alfa Aesar*, water suspension, 100 nm diameter) used as a
106 catalyst for the vapor-liquid-solid (VLS) mechanism.

107 *CdS NWs*: CdS powder (98%, *Alfa Aesar*) was loaded in a ceramic boat and
108 placed in the center of the quartz tube, Au/Si substrates were placed downstream in a
109 lower temperature region. The reactor was heated to 875°C under a flow of N₂ carrier
110 gas and maintained for 20 minutes for the vapor transport and NW growth, followed by
111 natural cooling down to room temperature.

112 *SnO₂ NWs*: 0.3g of SnO:C=2:1 powder mixture was loaded in a ceramic boat in
113 the center of the quartz tube at 880°C, the vapor was transported downstream to the
114 Au/Si substrate at a lower temperature region using N₂ as a carrier gas. The temperature
115 during the growth was held constant for 90 minutes, followed by a natural cooling down
116 to room temperature.

117 *ZnO NWs*: 1:1 mixture of ZnO and carbon powders was loaded in a quartz tube
118 and kept at 950°C temperature for 90 minutes, followed by natural cooling down to
119 room temperature. The vapor was carried downstream to the Au/Si substrate by N₂ gas.

120 The phase of as-grown NWs was determined by X-ray diffraction (XRD) using
121 Rigaku MiniFlex 600 X-ray powder diffractometer with Bragg-Brentano θ -2 θ
122 geometry and the 600W Cu anode (Cu K α radiation, $\lambda = 1.5406 \text{ \AA}$) X-ray tube. NW
123 morphology was characterized using a scanning electron microscope (SEM, Lyra,
124 Tescan), while their inner crystalline structure was revealed using a transmission
125 electron microscope (TEM, Tecnai GF20, FEI) operated at a 200 kV accelerating
126 voltage.

127

128 2.2 Single nanowire two-terminal photodetector device fabrication and measurements

129 Two-terminal individual NW-based photodetectors were fabricated via the
130 conventional photolithography technique. First, NWs were mechanically transferred to
131 an oxidized silicon wafer (50 nm thick SiO₂), followed by spin-coating it with AZ1518
132 photoresist. The optical mask with the microelectrode pattern (5 μm gap between the
133 electrodes) was aligned on top of a selected NW and exposed using the SUSS MicroTec
134 MA/BA6 Gen4 mask aligner. A 10/100/70 nm thick Cr/Ag/Al film, respectively, was
135 deposited via thermal evaporation method followed by a lift-off procedure. At least five
136 NW-based photodetectors for each material were fabricated so consistent conclusions
137 could be made.

138 Current–voltage (I – V) characteristics and photoresponse of the fabricated
139 individual NW-based photodetector devices were measured with a low-noise current
140 preamplifier (SR570, Stanford Research Systems) and oscilloscope (TDS2004B,
141 Tektronix). Conventional water-cooled tungsten anode X-ray tube operated within 10–
142 30 kV and 10–30 mA with 0.1 mm Be window was used as an X-ray source for NW
143 illumination providing estimated X-ray photon flux of 10⁹ (1/s·mm²). Spellman
144 XLF60N1200/230 power supply is used to ensure stable lamp operation. Since the tube
145 voltage was always below 30 kV, the K -characteristic lines of tungsten were never
146 excited, and the X-ray spectrum included contributions from the multiple L -emission
147 lines in the 7 keV–12 keV energy range and bremsstrahlung [30]. The schematic of the
148 experimental setup is depicted in *Fig.1*. A 405 nm wavelength semiconductor diode
149 laser (CNI Laser) with 0.5 W/cm² power was used for the optical photoresponse
150 measurements. All the measurements were performed under ambient conditions.

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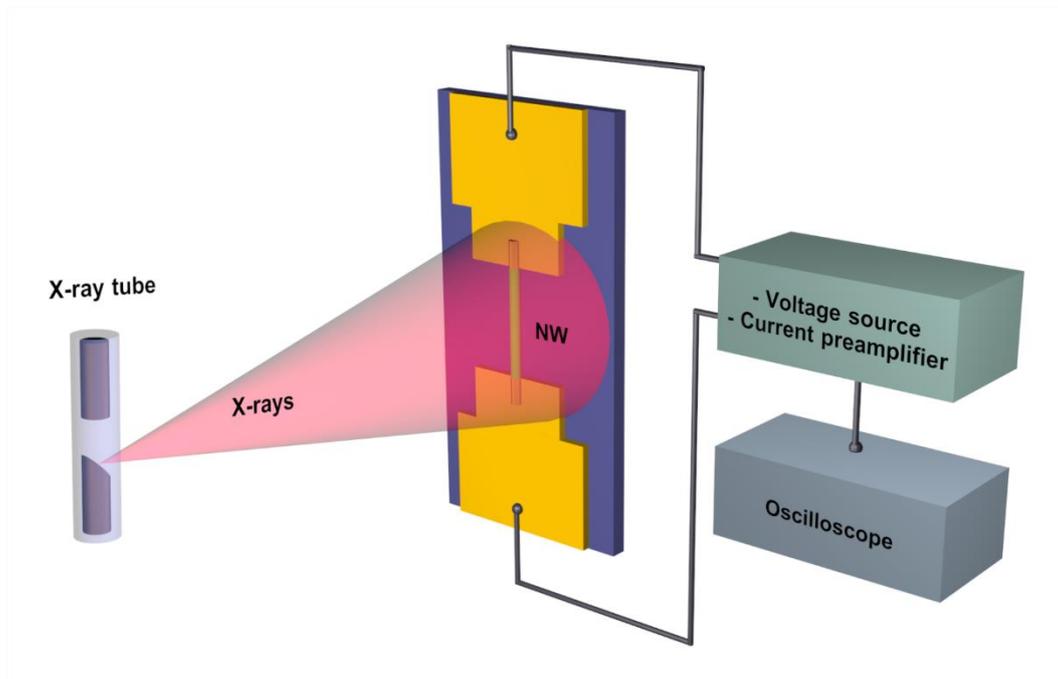


Figure 1. Schematic of the experimental setup. A single nanowire (NW) device on a Si/SiO₂ substrate was illuminated with X-rays, generated by a tungsten anode X-ray tube. X-ray beam induced current was measured by connecting the device to a low-noise current preamplifier and oscilloscope.

152

153 3. Results and discussion

154 The crystallographic structure and morphology of as-grown NWs are shown in
 155 *Fig. 2*. SEM images of CdS, SnO₂, and ZnO NW arrays (*Fig. 2(a-c)*, respectively) show
 156 smooth NWs with a length of 10 – 100 μm and a diameter of about 100 nm. To confirm
 157 the presence of the respective phases in the as-grown NW samples, XRD measurements
 158 were performed on the NW arrays on the Si(100)/SiO₂ substrates (see *Fig. 2(d)*). The
 159 XRD patterns of each sample contain Bragg peaks of the desired highly-crystalline
 160 phases: the pattern for CdS NWs was indexed to hexagonal wurtzite CdS structure
 161 (ICDD-PDF #41-1049), SnO₂ NWs exhibit tetragonal cassiterite SnO₂ structure
 162 (ICDD-PDF #41-1445), and the pattern of ZnO NWs corresponds to hexagonal wurtzite
 163 ZnO structure (ICDD-PDF #36-1451). The Bragg peak at $2\theta \approx 33^\circ$ is attributed to the
 164 Si(100) substrate (forbidden Si(200) reflection). Furthermore, the results of XRD

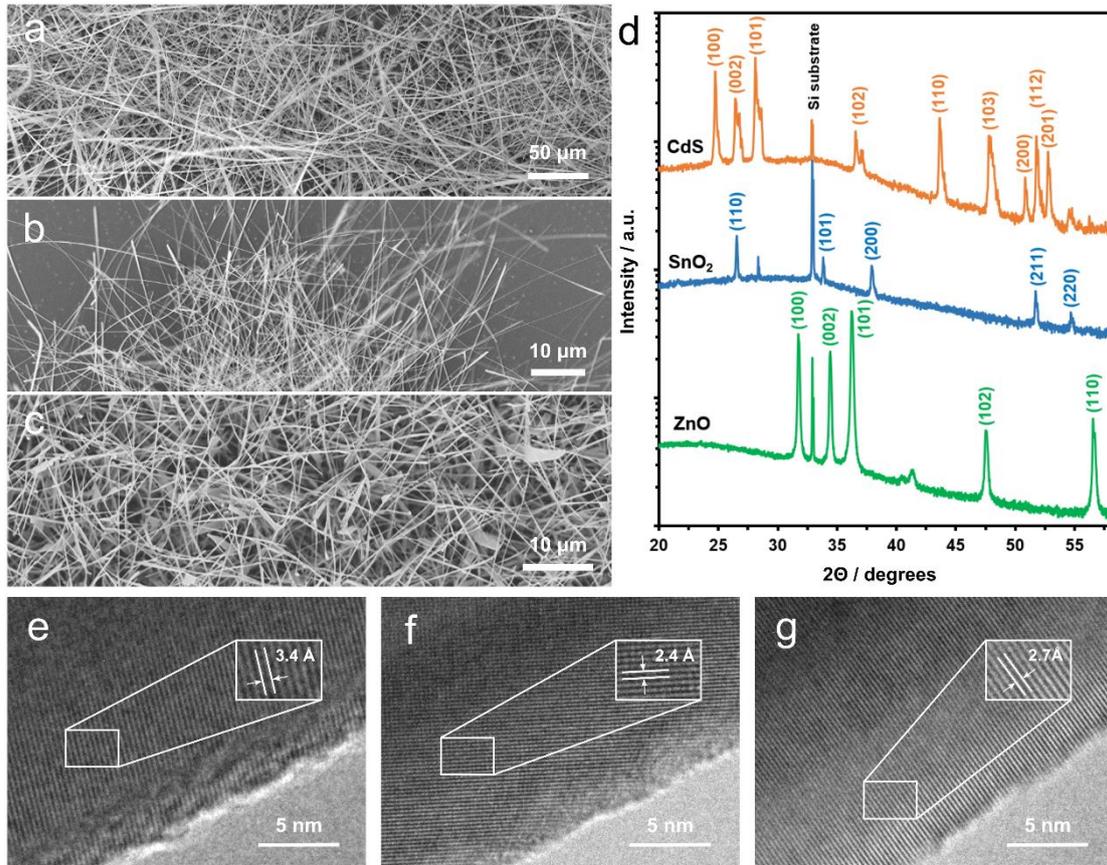


Figure 2. Scanning electron microscope images of (a) CdS, (b) SnO₂ and (c) ZnO nanowires. (d) X-ray diffraction patterns of CdS, SnO₂, and ZnO nanowire arrays on Si(100)/SiO₂ substrates. Transmission electron microscope images of (e) CdS, (f) SnO₂, and (g) ZnO nanowires; the insets show the measured d-spacings.

165 measurements and the high-quality single-crystalline nature of the NWs were
 166 confirmed by TEM studies (see *Fig. 2(e-g)*). Interplanar distances (d-spacings) in three
 167 materials NWs measured for the resolved atomic planes are 3.4 Å (for CdS), 2.4 Å (for
 168 SnO₂), and 2.7 Å (for ZnO) and are in good agreement with the lattice parameters given
 169 in the corresponding ICDD-PDF files of hexagonal CdS ($c=6.73$ Å), tetragonal SnO₂
 170 ($a=b=4.74$ Å) and hexagonal ZnO ($c=5.20$ Å).

171 The photoconductivity measurements were performed on two-terminal
 172 individual NW-based devices produced using NWs transferred to Si(100)/SiO₂
 173 substrates and conventional lithography technique. First, dark state current-voltage (I-
 174 V) characteristics of individual CdS, SnO₂, and ZnO NWs were measured (see *Fig.3*).

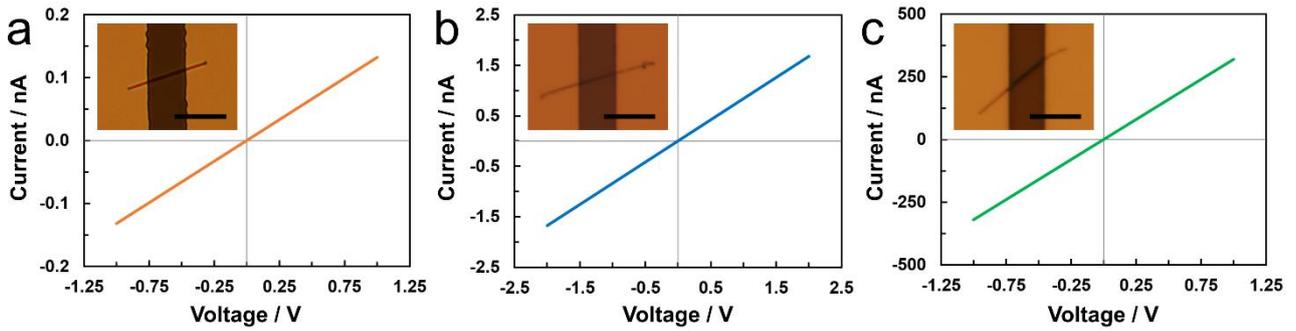


Figure 3. Dark state I-V characteristics of (a) CdS, (b) SnO₂, and (c) ZnO single-nanowire devices. Insets show optical microscope images of the nanowires lying on electrodes, scale bars correspond to 7 μm.

175 The I-V characteristics of all three materials exhibit linear behavior, indicating that the
 176 ohmic contacts were formed between the NWs and the electrodes (chromium bottom
 177 layer) [15,18,31], which is important for the efficient collection of photogenerated
 178 carriers. CdS NWs exhibit a small dark current around 100 pA, the typical value for
 179 SnO₂ NWs was around 1 nA, while ZnO NWs have a significantly higher dark current,
 180 of the order of hundreds of nanoamperes. Note that semiconductor-based X-ray sensors
 181 should have a low dark current to obtain a high signal-to-noise ratio, similarly to visible-
 182 light photodetectors.

183 Next, the fabricated NW-based devices were illuminated with laser light with a
 184 wavelength of 405 nm to check their photoconductive properties in the visible region
 185 of light. The results of the time-dependent photocurrent measurements and
 186 photodetector parameters are given in *Fig.S1* and *Table S1*. All NW-based devices
 187 demonstrate very high spectral responsivity (up to 1429 A/W for SnO₂ NWs) and
 188 external quantum efficiency with few μA large photocurrent, which is comparable to
 189 similar *state-of-the-art* individual NW-based photoconductors [17,18,32,33]. The
 190 response times (rise and decay) are around 150 ms for CdS and SnO₂ NWs, while ZnO
 191 NWs typically exhibit slow response in a range of several seconds [17]. The slow
 192 response speed and the high dark current of ZnO NWs can be altered by modifying

193 their surface, for example, with Au or PbI₂ which might also increase the X-ray
 194 absorption of the nanostructures [34,35]. The excellent photodetection performance
 195 (high photoconductive gain) indicates the high quality of the synthesized NWs and the
 196 fabricated devices.

197 The as-fabricated individual NW-based devices were periodically exposed to
 198 X-rays generated by a conventional tungsten anode X-ray tube at fixed tube voltage
 199 ($U_{\text{tube}}=30$ kV) and current ($I_{\text{tube}}=10$ mA). X-ray photon flux was estimated to be around
 200 10^9 (1/s·mm²). Fig.4(a-c) shows the measured on/off photocurrent curves in individual
 201 CdS, SnO₂, and ZnO NWs at different sample bias voltages. A stable increase of the
 202 current in response to the X-ray illumination has been detected in all three cases. The
 203 overshooting transient signal in Fig.4(a) can be attributed to charge trapping/de-
 204 trapping in semiconductor/electrode interfaces and to space-charge effects. Time-

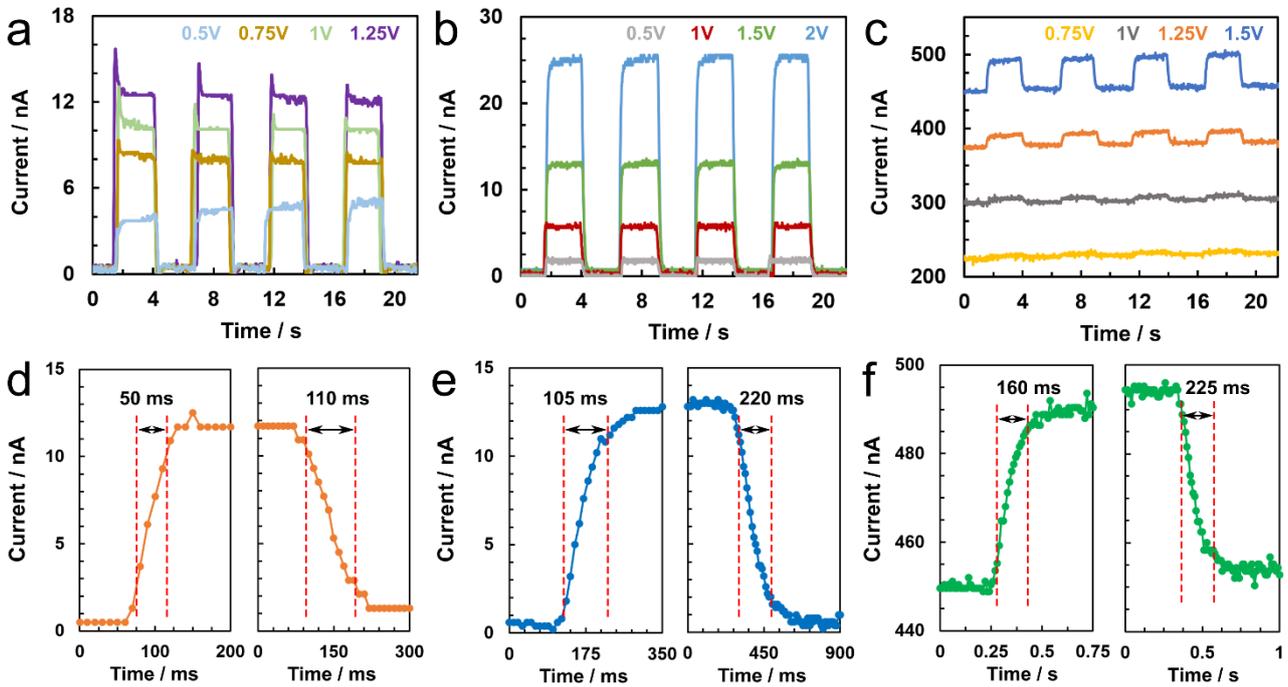


Figure 4. On-off photoresponse to X-ray illumination at fixed tungsten anode X-ray tube voltage (30 kV) and current (10 mA) but different bias voltages, and the respective time-resolved graphs for (a,d) CdS, (b,e) SnO₂, and (c,f) ZnO single-nanowire devices.

205 resolved graphs in *Fig.4(d-e)* show the rise and decay rates of XBIC, the values being
 206 in the range of 50-225 ms, which is sufficient for practical applications. The rise and
 207 decay times are defined as the required time for the photocurrent to increase or decrease
 208 to 90% or 10% of its maximum value, respectively. The magnitude of XBIC increases
 209 proportionally to the applied bias voltage since the photogenerated charge carriers can
 210 be collected more efficiently. High I_{on}/I_{off} ratios (up to around 100) were observed for
 211 CdS and SnO₂ NWs, while it was very small for ZnO NWs (up to 1.1) due to its high
 212 dark current. The measured XBIC is remarkably high, considering that the interaction
 213 volume is significantly smaller than in conventional thin film or bulk pixelated X-ray
 214 detectors. The high XBIC measured in the NW devices can mainly be attributed to the
 215 efficient transport and collection of generated charge carriers due to the single-
 216 crystalline nature of NWs and the short NW length. According to simplified
 217 calculations of X-ray induced steady state conductance similar to Wallentin *et al.* [11]:
 218 $G=q\mu N/l^2$, where G – conductance ($G=\Delta I/U$), q – electron charge, l – NW length, μ –
 219 electron mobility, N – number of free electrons. For SnO₂ mobility $\mu=100$ cm²/Vs (CdS
 220 $\mu=87$ cm²/Vs, ZnO $\mu=220$ cm²/Vs), $l=5 \cdot 10^{-4}$ cm, $\Delta I =10$ nA, $U=1$ V. For $G=10^{-8}$ Ω^{-1}
 221 we have $N=156$ electrons. On the other hand, number of X-ray induced steady state
 222 carriers $N=\gamma\tau$, where γ - is carrier generation rate and τ - recombination lifetime. Carrier
 223 generation rate $\gamma= p_{abs}\eta\Phi$, where p_{abs} – X-ray photon absorption probability, Φ - X-ray
 224 flux, and η - number of electron-hole pairs generated per X-ray photon. If we assume
 225 $p_{abs}\approx 10^{-4}$, $\eta\approx 10^4$, and $\Phi=1700$ ph/s, the carrier generation rate is $\gamma=1700$ s⁻¹,
 226 recombination lifetime is $\tau\approx 10^{-1}$ s, then we obtain $N=170$ electrons, which is close to
 227 the experimental value. Other effects, which arise from the NW size, such as charge
 228 separation due to surface traps, photogating (influence of substrate) or photodoping
 229 [11], might play a role in the process, however, the significance of these factors should

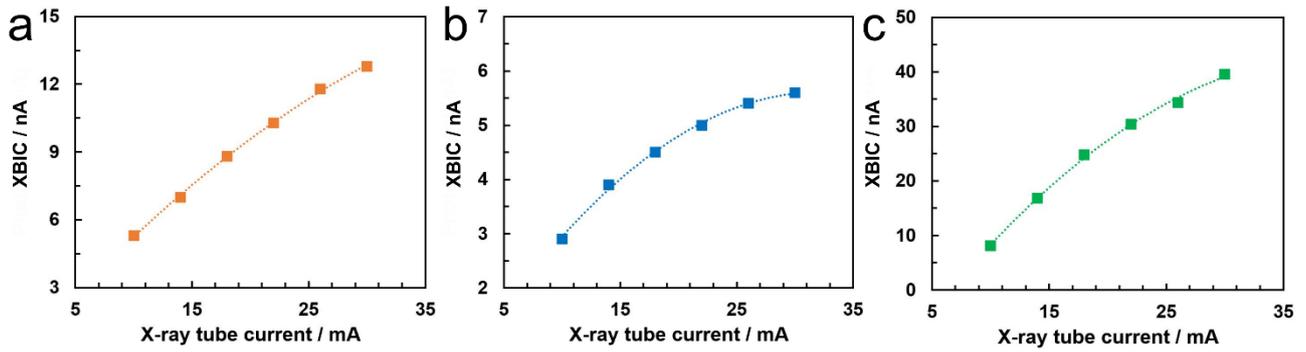


Figure 5. X-ray beam induced current (XBIC) dependence on X-ray tube current at fixed bias (1 V for CdS, SnO₂ and 1.5 V for ZnO) and tube voltage (10 kV) for (a) CdS, (b) SnO₂, and (c) ZnO single-nanowire devices. The points are experimentally measured values and the dashed lines have been added for visual clarity.

230 be elucidated. Furthermore, no detectable temporary or permanent degradation of the
 231 XBIC due to the exposure to X-rays over periods of several minutes was observed in
 232 the NWs, demonstrating the stability needed for practical applications. Finally, since
 233 relatively large areas of the samples were exposed to X-rays during the experiments, an
 234 equivalent microelectrode pattern without any NW was also tested for comparison. An
 235 electric signal around 100 pA was detected, which could be attributed to photoelectrons
 236 emitted from the metal contacts [26] and leakage current through ionized substrate/air
 237 gap; however, this signal does not contribute significantly to XBIC in NW-based
 238 devices since it is orders of magnitudes smaller.

239 XBIC dependence on the X-ray tube current and voltage was also measured.
 240 During the measurements, the current of the X-ray tube was varied between 10 mA and
 241 30 mA, while the voltage ($V_{\text{tube}}=10$ kV) and device bias ($U_{\text{bias}}=1$ V for CdS, SnO₂ and
 242 1.5 V for ZnO NWs) were kept constant. It can be seen in *Fig.5* that XBIC increases
 243 strongly with the tube current in a sub-linear fashion. A similar relation was observed
 244 when changing the tube voltage (see *Fig.S2*). Generally, an increase of the X-ray tube
 245 current or voltage results in larger X-ray photon flux [30], i.e., more X-ray photons are
 246 being generated, which leads to more absorbed photons in the NW in a unit of time,

247 subsequently giving larger XBIC. At the same time, when the voltage on the tube
248 changes, X-ray photons with higher energy are additionally generated, therefore, more
249 secondary electron-hole pairs are created per absorbed photon (higher yield of
250 photogenerated electron-hole pairs per X-ray photon), which again leads to a higher
251 XBIC. The reason for the sublinear dependence in *Fig.5* might be related to a decrease
252 in the charge carrier collection efficiency, since the high density of photogenerated
253 carriers at higher photon energies may influence the electrostatic potential distribution
254 in the NWs [26].

255

256 **4. Conclusions**

257 In this study, we demonstrated X-ray detection using individual CdS, SnO₂ and
258 ZnO nanowires, which were produced via vapor-liquid-solid technique and
259 characterized using X-ray diffraction, scanning and transmission electron microscopy.
260 Photocurrent measurements were performed under ambient conditions while exposing
261 two-terminal individual NW-based devices to X-rays generated by a conventional
262 tungsten anode X-ray tube.

263 X-ray beam induced current of tens of nanoamperes and a response rate in the
264 millisecond range were measured in the individual NWs, which is remarkably high,
265 considering that the interaction volume of NWs is significantly smaller than in
266 conventional thin film or bulk pixelated X-ray detectors. The high X-ray beam induced
267 current could mainly be attributed to the efficient transport and collection of generated
268 charge carriers due to the single-crystalline nature of NWs and the short NW length.
269 While X-ray detectors based on CdS and SnO₂ NWs exhibited a small dark current and
270 high on/off current ratio, a high dark current was measured in ZnO NWs, which limits
271 their applicability, however, that might be solved by modifying ZnO NW surface states.

272 Our study demonstrates that the possibilities of detecting X-rays using various
273 NWs and NW-based heterostructures containing high-Z element materials merit a more
274 thorough investigation. In the future, semiconductor NWs may enable the production
275 of novel high-resolution X-ray detectors, suitable for both synchrotron radiation,
276 especially nanofocused beams, and conventional X-ray tubes.

277

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288 **Supplementary information**

289 Supplementary information is available and contains CdS, SnO₂, and ZnO
290 individual NW-based photodetector response measurements to visible (405 nm) light,
291 as well as a table containing spectral responsivity and external quantum efficiency
292 calculated for these devices. These same devices were used for the XBIC measurements
293 reported in the main text. XBIC dependence on X-ray tube voltage is also shown.

294

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