Surface Treatments and Photodesorption of Oxygen Free Copper used in an Accelerator

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Abstract
Test samples of machined oxygen free copper (OFC) were exposed to synchrotron radiation from the Photon Factory (PF) of KEK for a critical energy of 4 keV. In experiments, we measured the photoelectron yield and the photodesorption yield due to synchrotron radiation. The irradiated surfaces were also analyzed by AES.

I. INTRODUCTION
Oxygen Free Copper (OFC) has good properties as the material for vacuum components in an accelerator. Such properties include high conductivity and good shield characteristics that are effective against high energy radiation. OFC also shows lower photodesorption yields[13]. Photodesorption in accelerators is affected by the photon energy, the photon flux, and the surface treatments, etc. Some studies focusing on a photodesorption process[4-10] have shown that photoelectrons emitted from irradiated surfaces are the main cause of photodesorption.

The purpose of this experiment is to study the effects of irradiation of synchrotron radiation on OFC in terms of photoelectron production, surface changes, and desorption. While OFC samples are irradiated by synchrotron radiation photocurrent and desorption are measured. The irradiated surfaces are also analyzed by AES.

II. EXPERIMENT
A. Experimental setup

The apparatus at the BL21 of PF is reported in detail in Reference[8]. The experimental set up for this experiment is shown in Figure 1. Synchrotron radiation from the PF ring enters the chamber after being collimated at the slit. The size of the photon beam is 5 mm in both directions. The slit also works as an orifice of 3 l/s conductance (N2 equivalent).

OFC samples (max. of four) are first attached to the sample holder. The sample holder is then set in a chamber made of stainless steel through a ceramic chamber, the sample holder is floating. At this point, we can measure the photocurrent produced in samples. A photoelectron stopper made of OFC in front of the irradiated samples to possibly reduce the desorption from the chamber due to irradiation of secondary particles, such as secondary electrons and reflected photons.

B. OFC samples

All samples are made of high-purity oxygen free copper (ASTM-F68 Class-I)[11]. Table 1 lists the samples. Except for Sample 1-2 (extruded), the average roughness (Ra) of a machine finished[1] samples is either Ra=12.5 μm or 0.5 μm. Acetone is used for degreasing with ultrasonic agitation for 30 min. HNO3 of 45% concentration by volume (from HNO3 of 65% concentration by weight) is used for acid cleaning, after acid cleaning deionized water is used for rinsing, methanol and dry nitrogen are used for drying. There are two series of experiments: one series is indicated by "Sample 1-" and the other is "Sample 2-". The samples were irradiated serially in each series without exposure to air.

Table 1. OFC samples

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Roughness(Ra:μm)</th>
<th>Treatment</th>
<th>Dose(mA*h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>12.5 (machining)</td>
<td>degreasing</td>
<td>0</td>
</tr>
<tr>
<td>1-1</td>
<td>12.5 (machining)</td>
<td>degreasing</td>
<td>50</td>
</tr>
<tr>
<td>1-2</td>
<td>raw (extrusion)</td>
<td>degreasing</td>
<td>6,090</td>
</tr>
<tr>
<td>1-3</td>
<td>12.5 (machining)</td>
<td>degreasing</td>
<td>1,020</td>
</tr>
<tr>
<td>1-4</td>
<td>12.5 (machining)</td>
<td>degreasing</td>
<td>4,880</td>
</tr>
<tr>
<td>2-1</td>
<td>0.1 (machining)</td>
<td>degreasing</td>
<td>790</td>
</tr>
<tr>
<td>2-2</td>
<td>0.1 (machining)</td>
<td>degreasing</td>
<td>12,590</td>
</tr>
<tr>
<td>2-3</td>
<td>0.1 (machining)</td>
<td>acid cleaning</td>
<td>27,340</td>
</tr>
<tr>
<td>2-4</td>
<td>12.5 (machining)</td>
<td>acid cleaning</td>
<td>49,390</td>
</tr>
</tbody>
</table>

III. RESULTS AND DISCUSSION
A. Photoelectron yield

Figure 2 and Figure 3 show photoelectron yields in the first series and in the second series, respectively. The horizontal axis represents the integrated photon dose which is initialized after each direct irradiation on to a sample. A beam dose of 1 mA*h
is equivalent to a cumulated photon dose of $1.75 \times 10^{17}$ photons/slit introduced into the chamber through the slit, the order of irradiation is serial from Sample 1-1 through Sample 1-4. Note that degreasing is a common treatment in Figure 2. The photoelectron yields are similar for Sample 1-3 and Sample 1-4; these samples are machine finished and have the same roughness. Sample 1-2 with an extruded surface has a smaller yield. Due to a data acquisition system error, the photoelectron yields in Figure 2 are shown from a point midway through irradiation of Sample 1-2. The difference in photoelectron yields is attributed to the difference in surface conditions. It is considered that Sample 1-2 still contains an impurity giving the lower photoelectron yield, e.g., carbon produced in the extrusion process.

![Photoelectron yield in the first experimental series as a function of direct photon dose.](image1.png)

The photoelectron yields in the second series are shown in Figure 3. The average roughness for Samples 2-1, 2-2, and 2-3 is 0.1 μm and that of Sample 2-4 is 12.5 μm. Samples 2-1 and 2-2 are degreased with acetone, and Samples 2-3 and 2-4 are treated with HNO₃. The tendency of decreasing photoelectron yields with increasing photon dose for Samples 2-1 and 2-2 is similar to that for Samples 1-3 and 1-4 in Figure 2. The common factor here is acetone degreasing. However, the yields for Samples 2-1 and 2-2, each with average roughness 0.1 μm, are smaller than those of Samples 1-3 and 1-4.

The decreasing tendency for the samples treated with HNO₃ differs from that for Samples 2-1 and 2-2, at photon doses higher than $10^{19}$ photons. Samples 2-3 and 2-4 experience a smaller slope. The yield of Sample 2-3 begins decreasing again like the yield of the Sample 2-2, but the yield of Sample 2-3 is higher than that of Sample 2-2. The yield of Sample 2-4 is almost constant and maintains a high value. It is presumed that these behaviors of photoelectron yields for samples treated with HNO₃ are due to an oxide layer produced in the acid cleaning stage. This oxide layer possibly increases the photoelectron yields. The difference in yields between Samples 2-3 and 2-4 at photon doses higher than $10^{21}$ photons means that surface roughness affects the oxide layer production in the acid cleaning stage. The total dose, i.e., the integrated dose from the beginning of the experiment, is different for each irradiation. In spite of this difference, the yields are not so different among the samples at lower photon dose. This means that photoelectron yield is not clearly influenced by the total dose.

In comparison with the photoelectron yield of aluminum alloy [9] at $2 \times 10^{21}$ photons under normal incidence, the yield of Sample 2-2 is almost one and half times higher.

![Photoelectron yield in the second experimental series as a function of direct photon dose.](image2.png)

**B. Surface analysis**

The surface concentrations of the machine finished samples in the first experiment are shown in Figure 4. These results are obtained from AES analysis. The term "Others" includes Cl, S, N impurities. Sample 0 was not exposed to synchrotron radiation. The carbon ratio decreases for a small dose (Sample 1-1), but increases with increasing in photon dose (Samples 1-3 and 1-4). Consequently, the decrease in photoelectron yield with increasing photon dose in Figure 2 is probably caused by this carbon ratio increase on the surface. The ratio for carbon of Sample 1-3 to Sample 1-4 is almost same as the ratio for photoelectron yield of Sample 1-4 to Sample 1-3 at the last photon dose respectively. The pressures at this conditions were $10^4$ Torr range.

![Surface concentration of Carbon, Oxygen, and Copper by AES analysis.](image3.png)

Figure 5 shows Auger depth profiles obtained by 3 keV argon ion sputtering. The sputtering rate is 120 Å/min (SiO₂ equivalent). The oxide layer becomes thick after photon irradiation.
C. Photodesorption yield

Figure 6 shows the photodesorption yield (total photodesorption yield: \(N_\text{eqi.}\)) in the second series. As shown in Figure 1, the size of samples is small and the area of the chamber inner surface is more than 50 times larger than that of the samples. Therefore, this photodesorption yield includes the effects of photodesorption from other components. Figure 6 also shows other yields\[^1\] measured using a test duct. The total photodesorption yield in the second series is close to the yield of stainless steel duct (SUS duct); it is slightly higher due to a complicated room within the chamber and a large surface area; in spite of these conditions, however, the yields are not so high. It is assumed that the high density region of photoelectrons is surrounded by the samples and stopper made of OFC.

IV. CONCLUSION

Irradiation of synchrotron radiation to OFC increases the carbon concentration on the irradiated surface. The photoelectron yield which affects photodesorption is changed by the photon dose and the surface treatment; surfaces with acid cleaning in particular maintain a high yield.

Acknowledgement

The authors are indebted to Dr. Kanazawa (KEK) for supplying the extruded OFC sample.

V. REFERENCES