

## Photoelectron Spectroscopy of Copper Tellurite Glass Surfaces

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**ABSTRACT.** Tellurite glasses containing CuO with the nominal composition  $(\text{CuO})_x (\text{TeO}_2)_{1-x}$ , where  $x = 0.30, 0.40$  and  $0.50$ , have been prepared and investigated by X-ray photoelectron spectroscopy (XPS). The Spectra of the core levels Cu 2p, Te 3d, and O 1s in these glasses have been measured. The appearance of a satellite peak in the Cu 2p core level spectra provides definitive evidence for the presence of  $\text{Cu}^{2+}$  ions in these glass samples where the asymmetry and broadening of the peaks are indicative of the presence of both  $\text{Cu}^{2+}$  and  $\text{Cu}^+$  ions. The broadened Cu 2p<sub>3/2</sub> peaks were decomposed into two distinct peaks separated by approximately 2.0 eV, with the lower energy peak being associated with  $\text{Cu}^+$  and the higher energy peak with  $\text{Cu}^{2+}$ . The relative  $\text{Cu}^{2+}$  content determined from the relative areas under these peaks and the satellite peak is found to vary from 40% for the  $x = 0.50$  glass sample to over 90% for the  $x = 0.30$  sample. The O 1s spectra for all values of  $x$  show slight asymmetry and were therefore fitted with two contributions, one from bridging oxygen (BO) and the other from non-bridging oxygen (NBO). The BO signal is due to the presence of oxygen atoms in the environment Te-O-Te, while the NBO is due to oxygen atoms in the environment Te-O-Cu. The ratio NBO/total oxygen was evaluated for each glass and was found to be in good agreement with the theoretical values calculated from the glass composition. The Te 3d core level spectra show symmetry for all values of  $x$  and indicate that the chemical environment of Te atoms does not vary much with  $x$  values.

### 1. Introduction

Studies of semiconducting oxide glasses containing transition metal (TM) ions continue to be of technological interest because of their increasing applications [1] in different fields. Tellurite glasses, a distinct type of non-crystalline materials, having unique structure and properties are also of special interest in view of their applications [2] in IR domes, laser windows and multifunctional optical components. From various spectroscopic methods such as infrared [3-5], Raman [5-9], nuclear magnetic resonance [10], and X-ray absorption spectroscopy [11-13] as well as from X-ray [13-14] and neutron diffraction [15-

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16] techniques, it has been established that the basic structure of the tellurite glasses is a  $\text{TeO}_4$  trigonal bipyramid (tbp) with a lone pair of electrons in one of its equatorial sites. Tellurium oxide under normal conditions does not have the ability to form a glass structure easily without a modifier like an alkali oxide, an alkaline-earth oxide, a transition-metal oxide, or another glass former [15,17-18]. Researchers have different opinions as to why it is so difficult to form a glass of pure  $\text{TeO}_2$ . Neov *et al.* [15,17] hypothesized that  $\text{TeO}_2$  could not form a glass by itself because the Te-O bond is too strongly covalent to permit the requisite amount of distortion for a glass structure. Another prevalent view [19-20] is that the repulsive forces due to the lone pair of electrons resist the free movement of the trigonal bipyramid in space during the cooling of the melt and hence the formation of glass. In a binary tellurite glass the effect of the lone pair of electrons is limited by the introduction of new structural units that are compatible with  $\text{TeO}_4$  (tbp) and thus the glass formation becomes easier. Most of the tellurium atoms of  $\text{TeO}_4$  structure are connected at vertices by a  $\text{Te}_{\text{eq}}\text{O}_{\text{ax}} - \text{Te}$  bond, a prerequisite for glass formation, where the symbol " $\text{eqO}$ " refers to an oxygen in an equatorial plane and " $\text{O}_{\text{ax}}$ " refers to the oxygen in an axial position with respect to the Te atom [15,17]. These structural peculiarities may be reflected in various physical properties of these glasses making them interesting materials for study in themselves.

The addition of transition-metal (TM) oxides to glasses, in general, permits the possibility for the glasses to exhibit both semiconducting and magnetic properties. The general condition for semiconducting behavior in these glasses is that the TM ion must be capable of existing in more than one valence state so that conduction can occur by the electron transfer from ions in a lower valence state to those in a higher valence state [21]. Structural and electronic properties of these glasses as well as their optical, magnetic, and mechanical properties depend on the relative proportion of the different valence states of the TM ions present [22-24]. In order to account for the effect of these valence states on the structure and properties of these glasses, it is important to control and measure the ratios of the TM ion concentration in the different valence states of these oxide glasses. In previous studies [25-28], electron paramagnetic resonance (EPR) has been found to be a useful technique for examining the states of dissimilar TM ions in these glasses as well as the interactions between unlike magnetic ions in disordered solids. Alternatively, X-ray photoelectron spectroscopy (XPS) has been found [29] to be a useful technique in assessing the local glass structure and even estimating the ratio of the different valence states in the TM-oxide glasses. The technique has been successfully used to distinguish between bridging oxygen (BO) and non bridging oxygen (NBO) [30]. In this study, we will use XPS to investigate the role of Cu in Cu-tellurite glasses. Recently ultrasonic study has been carried out [31] on this system but no XPS studies have been reported in the literature. We will record Te 3d, Cu 2p and O 1s spectra to try to identify the possible Te environment, namely  $\text{TeO}_3$  and  $\text{TeO}_4$  as pointed out in the literature, estimating the different valence states of Cu, and measuring the concentration of possible NBO atoms to BO atoms in these glasses.

## 1. Experimental Procedure

All glasses were prepared by melting dry mixtures of reagent grade CuO, and  $\text{TeO}_2$  in alumina crucibles to form batch  $(\text{CuO})_x (\text{TeO}_2)_{1-x}$  compositions with  $x = 0.3, 0.4$  and  $0.5$ . Approximately 30g of chemicals were thoroughly mixed to obtain a homogenized mixture for each CuO concentration. The crucible containing the batch mixture was then

transferred to an electrically heated melting furnace maintained at 800-850 °C. The melt was left for about an hour under atmospheric conditions in the furnace during which the melt was occasionally stirred with an alumina rod. The actual compositions of the glasses were determined by inductively coupled plasma spectroscopy (ICP) and are listed in Table 1.

Table 1. Batch and actual composition (molar fraction) of various tellurite glasses containing CuO.

x	Batch		Actual (from ICP)	
	TeO <sub>2</sub>	CuO	TeO <sub>2</sub>	CuO
0.30	0.70	0.30	0.728	0.272
0.40	0.60	0.40	0.643	0.357
0.50	0.50	0.50	0.509	0.491

High-resolution photoelectron spectra were collected on a VG scientific ESCALAB MKII spectrometer equipped with dual aluminum-magnesium anode X-ray gun and a 150 mm concentric hemispherical analyzer using Al K $\alpha$  radiation ( $h\nu = 1486.6$  eV) radiation from an anode operated at 130 W [32]. Photoelectron spectra of Te 3d, Cu 2p, and O 1s core levels were recorded using a computer controlled data collection system. The electron analyzer was set at a pass energy of 20 eV. For self-consistency, the C 1s line at a binding energy of 284.6 eV was used as a reference for all charge shift corrections as this peak arises from hydrocarbon contamination and its binding energy is generally accepted as remaining constant, irrespective of the chemical state of the sample. For XPS measurements, a glass rod from each composition was cleaved in the analysis chamber at a base pressure  $2 \times 10^{-9}$  mbar before being transferred to the analysis chamber where the pressure was maintained at  $< 2 \times 10^{-10}$  mbar.

## 2. Results and Discussion

Relatively low-resolution X-ray (0.5 eV step-size) photoelectron scans, taken from the fractured surfaces, in the binding energy range of 0 - 1200 eV were obtained for each glass sample in about one hour using Al K $\alpha$  incident photons with Fig. 1 showing wide scan spectra for some compositions. Each spectrum includes the photoelectron and X-ray induced Auger peaks of the glass constituents. The observed C 1s peak at 284.6 eV is associated with hydrocarbon contamination and is typically present on samples introduced from the laboratory environment. Thus it was used as an energy reference for determining peak positions for the Te 3d, Cu 2p, and O 1s core levels as listed in Table 2.

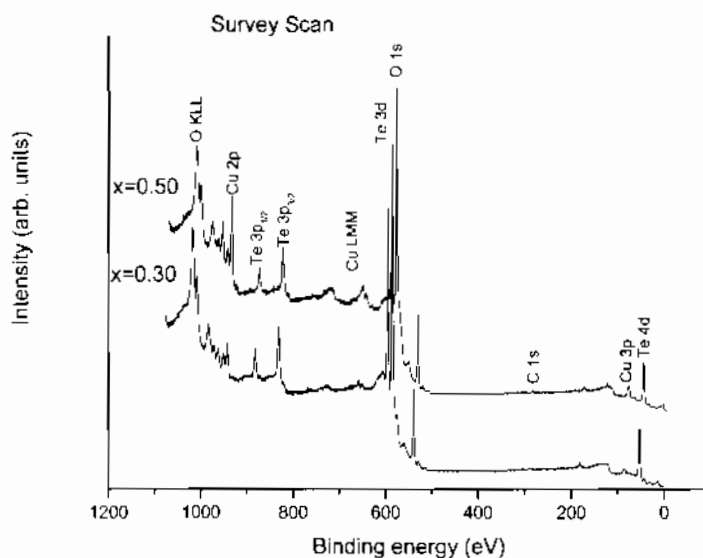


Fig. 1. Wide scan XPS spectra from the surface of copper-tellurite glasses obtained using Al K $\alpha$  radiation ( $h\nu = 1486.6$  eV) for the  $x = 0.30$  and  $x = 0.50$  glasses.

Table 2. Peak positions in eV for the core levels Te 3d, Cu 2p and O 1s relative to C 1s (284.6 eV) and their corresponding FWHM (full-width at half maximum). The uncertainty in the peak position is  $\pm 0.10$  eV and in FWHM is  $\pm 0.20$  eV.

x	Te 3d <sub>5/2</sub>	Te 3d <sub>3/2</sub>	$\Delta E$ Te3d	Cu 2p <sub>3/2</sub>	Cu 2p <sub>1/2</sub>	$\Delta E$ Cu2p	O 1s
	FWHM	FWHM		FWHM	FWHM		FWHM
0.30	576.2	586.6	10.4	933.4	953.0	19.6	529.7
	2.47	2.45		4.10	4.60		2.44
0.40	575.9	586.3	10.4	933.4	953.4	20.0	529.7
	2.36	2.20		3.98	4.40		2.33
0.50	575.9	586.2	10.3	932.0	951.8	19.8	529.8
	2.60	2.62		3.34	3.65		1.91
TeO <sub>2</sub>	576.1	586.5	10.4				530.3
	2.07	1.96					1.93
CuO				933.6	953.5	19.9	529.5
				3.31	3.53		1.89

The Te 3d core level peaks shown in Fig.2 have been normalized to the height of the  $x = 0.5$  peak. The doublet peaks attributed to Te 3d<sub>3/2</sub> and Te 3d<sub>5/2</sub> in the Te 3d spectra have essentially the same binding energies for all glass samples as well as for the TeO<sub>2</sub> powder, independent of the CuO content.

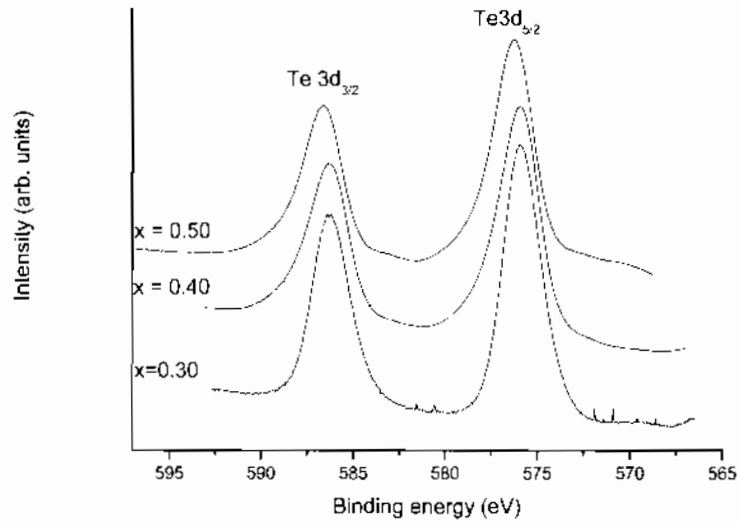


Fig. 2. Te 3d core level spectra for the copper-tellurite glasses.

Figure 3 shows the normalized core level spectra of Cu 2p for the glasses based on the height of the  $x = 0.5$  peak. The spectra exhibit spin-orbit components, Cu 2p<sub>3/2</sub> and Cu 2p<sub>1/2</sub>, at approximately 933 eV and 953 eV, respectively, with satellites at about 10 eV higher binding energies. Moreover, the Cu 2p peak shows a shoulder on the high binding energy side of the main contribution for the  $x = 0.5$  glass, while the shoulder is on the lower binding energy side for the other glass compositions. It is, therefore, more realistic to assume the presence of two separate contributions to the main Cu 2p peaks. These contributions are associated with monovalent (Cu<sup>1</sup>) and divalent (Cu<sup>2+</sup>) copper ions as these are the only two oxidation states in which Cu exists in various glasses [29]. It is well known that copper compounds containing Cu<sup>2+</sup> have strong satellite peaks as evident in the CuO spectra (labeled as satellite), while compounds with just Cu<sup>+</sup> have no satellites [33-36]. Hence the appearance of the satellite peaks can be used as a fingerprint for identifying the presence of Cu<sup>2+</sup> and so strong satellite peak in the Cu 2p core level spectra provides definitive evidence for the presence of Cu<sup>2+</sup> ions in these glass samples.

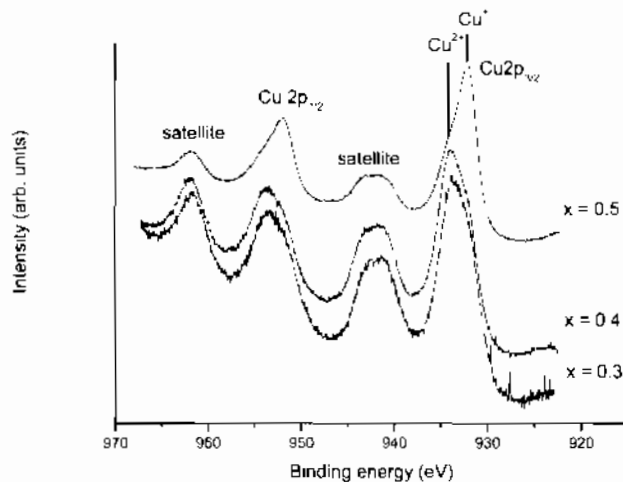


Fig. 3. Cu 2p spectra for the copper-tellurite glasses.

Assuming that the Cu 2p<sub>3/2</sub> spectrum can be composed of two overlapping peaks, each Cu 2p<sub>3/2</sub> spectrum was fitted to a sum of weighted Lorentzian-Gaussian peaks with a linear sloping background by means of a least squares fitting program. An example of the resulting fit for the x = 0.5 sample is shown in Fig.4. As previously reported [37], the areas under these peaks plus the area under the satellite peak can be used to determine the ratio of Cu<sup>2+</sup> ions to total Cu ions present.

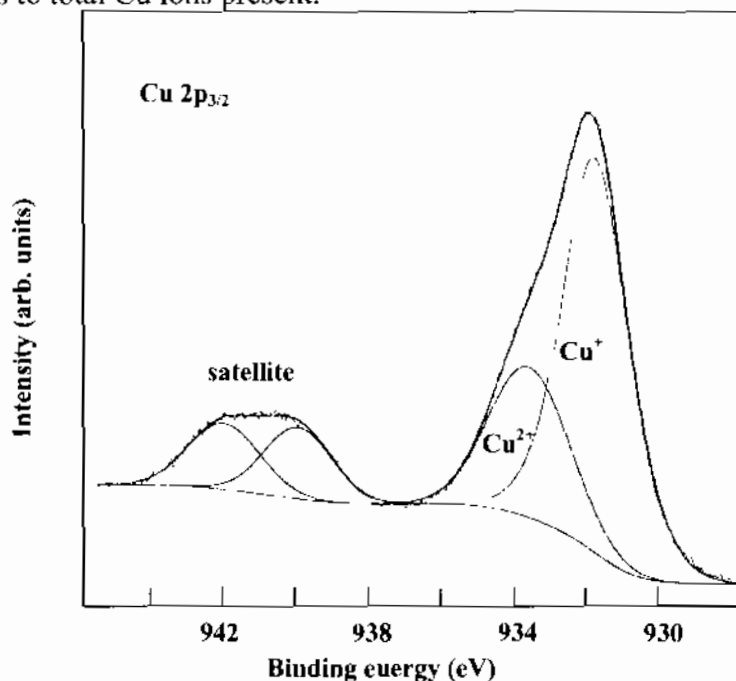


Fig. 4. Curve fitting of the Cu 2p<sub>3/2</sub> spectrum for a copper-tellurite glass (x = 0.50). The satellite peak has been fitted to two curves in order to accurately calculate the area under the peak.

#### Defining

concentration of Cu<sup>+</sup>  $\propto$  area under peak Cu<sup>+</sup> = A1

concentration of Cu<sup>2+</sup>  $\propto$  area under peak Cu<sup>2+</sup> + area under satellite peak = A2,

then, the concentration of Cu<sup>2+</sup> ions in each glass sample can be calculated as

$$\text{Cu}^{2+} / (\text{Cu}^{+} + \text{Cu}^{2+}) = \text{A2} / (\text{A1} + \text{A2}) \quad (\text{Eq. 1})$$

with the corresponding ratios displayed in Table 3 (The satellite has been fitted to two curves in order to accurately calculate the area under the satellite peak). This procedure should yield ratios with an estimated uncertainty of about 3%.

Table 3. Peak positions in eV from the curve fitting of the Cu 2p<sub>3/2</sub> peak and the ratio Cu<sup>2+</sup> / (Cu<sup>+</sup> + Cu<sup>2+</sup>) = A2 / (A1 + A2).

x	Cu 2p <sub>3/2</sub> (1) FWHM Peak Area	Cu 2p <sub>3/2</sub> (2) FWHM Peak Area	$\Delta$ Cu 2p	Cu 2p <sub>3/2</sub> (Sat) Peak Area	Cu <sup>2+</sup> /Cu <sub>total</sub> ( $\pm 0.030$ )
0.30	931.9 2.53 7586	934.0 3.20 20952	2.1	9412+4173 (13585)	0.820
0.40	931.9 2.53 15218	933.8 2.90 35273	1.9	14739+9843 (24582)	0.797
0.50	931.9 2.40 42882	933.9 2.90 19189	2.0	7438+7694 (15132)	0.444

In Fig. 5, the normalized O 1s core level spectra are shown. The maximum intensities of the spectra are the same position for all glass samples but shifted by about 0.5 eV toward the lower binding energy compared to its value for TeO<sub>2</sub> powder. Further, a slight asymmetry in the O 1s core level peak is apparent, which indicates two different types of oxygen sites in these glasses. Hence, all O 1s spectra were fitted to two Gaussian-Lorentzian peaks in order to determine the peak positions and relative abundance of the different oxygen sites.

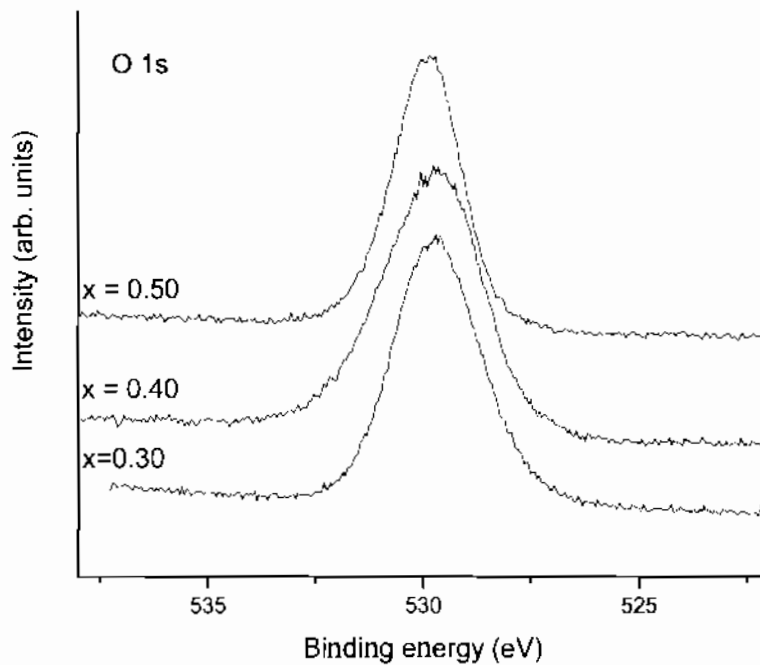


Fig. 5. The O 1s core level spectra for the copper-tellurite glasses.

As previously mentioned, the doublet peaks attributed to Te 3d<sub>3/2</sub> and Te 3d<sub>5/2</sub> in the Te 3d spectra have essentially the same binding energies for all glass samples independent of the CuO content with essentially no shift of the binding energies in comparison to their values in TeO<sub>2</sub> powder (Table 2). It is well known [35] that the presence of non-equivalent atoms of the same element in a solid gives rise to core level peaks with measurably different binding energies. This non-equivalence of atoms can result from a difference in the formal oxidation state and/or a difference in the molecular environment. The binding energy increases with an increase in the oxidation state of a metal atom. In the situation in which the formal oxidation state is the same, the general rule is that the core level binding energy of the metal atom increases as the electronegativity of attached atoms or group increases [38]. As has been mentioned in the introduction, it has been established that the basic structure of the tellurite glasses is a TeO<sub>4</sub> trigonal bipyramid (tbp) with a lone pair of electrons in one of its equatorial sites. Hence the oxidation state of Te practically remains the same and so no shift is expected. Further, as one finds the next-nearest neighbors in TeO<sub>2</sub> are all Te atoms which are replaced by Cu atoms in the TeO<sub>4</sub> glass structures and since the Pauling electronegativity of Cu (1.90) is almost the same as that of Te (2.1), suggesting practically no change in the binding energies of Te 3d spectra.

In most XPS studies of oxide glasses, the O 1s spectra are more informative with respect to the structure of the glass than the cation spectra. Specifically, the binding energy

of the O 1s electrons is a measure of the extent to which electrons are localized on the oxygen or in the internuclear region, a direct consequence of the nature of the bonding between the oxygen and different cations. As mentioned earlier, a slight asymmetry in the O 1s core level peak is apparent, which indicates two different types of oxygen sites in these glasses. If the O 1s peak for the glasses were composed of more than one component peak, they may correspond to the oxygen atoms in Te-O-Te and Te-O-Cu structural units. The oxygen atoms in Te-O-Te are termed "bridging" oxygens (BO) while those in Te-O-Cu are termed as "non-bridging" oxygens (NBO). Notice that the BO is covalently bonded to two glass former atoms while the NBO are ionically bonded at least from one side. Therefore, the binding energy of NBO is lower than that of BO. As clear from Fig. 5 and Table 2, the O 1s core-level spectra show composition-dependent changes; the FWHM (Table 2) increases with an increase of CuO content. It would indicate the presence of more than one contribution to the O 1s spectrum. Therefore, each O 1s spectrum was deconvoluted into two Lorentzian-Gaussian peaks with the lower binding energy peak corresponding to non-bridging oxygen atoms (Te-O-Cu) and the higher energy peak to bridging oxygen atoms (Te-O-Te) as shown in Fig. 6 for the  $x = 0.3$  glass sample. The ratio of the integrated areas of these peaks, NBO/Total oxygen, as summarized in Table 4, should represent the relative concentrations of bridging oxygen atoms. The ratio increases with an increase in the CuO concentration. It will be appropriate to mention that in the XPS study carried out on  $R_2O\text{-TeO}_2$  (R: Li, Na, K, Rb and Cs) using a fresh surface fractured in an ultra high vacuum ( $\approx 7 \times 10^{-8}$  Pa) only a single symmetrical Gaussian-Lorentzian peak was observed from which BO and NBO could not be separated [39].

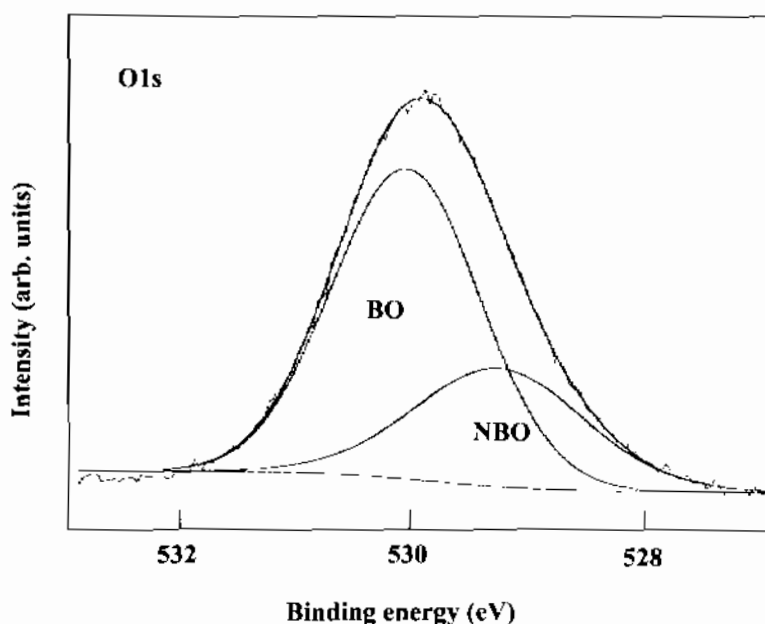


Fig. 6. Curve fitting of the The O 1s spectrum for a copper-tellurite glass ( $x = 0.30$ ) to two contributions.

If the copper atoms in the glass do indeed behave as network modifiers (introducing NBO), each  $\text{Cu}_2\text{O}$  and  $\text{CuO}$  molecule will contribute two NBO atoms in the glass [40]. Hence

$$\text{NBO/Total Oxygen} = [2 (\text{CuO}) + 2 (\text{Cu}_2\text{O})] / [ (\text{CuO}) + (\text{Cu}_2\text{O}) + 2 (\text{TeO}_2)] \quad (\text{Eq. 2})$$

where the terms in brackets indicate concentrations. There is a good agreement between the ratios of NBO/total oxygen obtained from the above equation and XPS analysis as shown in Table 4.



Table 4. Peak positions in eV from the curve fitting of the O 1s peak and the ratio NBO / (BO + NBO ) for Cu-Te glasses.

x	O1s (NBO) FWHM Peak Area	O 1s (BO) FWHM Peak Area	$\Delta E_{O1s}$	NBO/O <sub>total</sub> (Measured) ( $\pm 0.030$ )	NBO/O <sub>total</sub> (Calculated)
0.3	529.1 2.1 3427	529.9 2.0 7848	0.8	0.304	0.353
0.4	529.2 1.9 6706	530.1 2.0 7246	0.9	0.481	0.500
0.5	529.2 1.7 6295	530.1 1.9 3783	0.9	0.625	0.666

Hence it is clear that the O 1s signal from both Te-O-Cu(I) and Te-O-Cu(II) contribute to the NBO signal which would suggest that CuO enters the network as a glass modifier.

It is observed from Table 3, that copper ions exist predominately in the Cu<sup>2+</sup> state for all the Cu-Te glasses except for x = 0.5 glass where only 44% of the copper ions are in the Cu<sup>2+</sup> state. Further, the Cu2p<sub>3/2</sub> core level binding energy for the x = 0.5 glass is ~ 1 eV lower than its value for the other glass samples. The above observations are supported by the fact that the shift in binding energy may result not only from a change in the molecular environment but also from a reduction in the formal oxidation state of the transition metal ion [36].

We tried to grow glasses with composition x = 0.6 but could not succeed. For that composition the quenched melt would crystallize and therefore x = 0.6 would be outside the glass formation region.

#### 4. Conclusion

This XPS study of the binding energies associated with the Te 3d core levels for the Cu-tellurite glasses indicates that the local glass structure is similar to that of the TeO<sub>2</sub> glass former as essentially no energy shifts are found for these core levels. The asymmetry found in the Cu 2p<sub>3/2</sub> peak in combination with the presence of a satellite peak approximately 10 eV higher in energy provides evidence for the presence of Cu ions being in the Cu<sup>2+</sup> oxidation state as well as in the Cu<sup>+</sup> state. By decomposing the Cu 2p<sub>3/2</sub> into two distinct Lorentzian-Gaussian peaks separated by 2.1 eV with the higher energy peak being associated with Cu<sup>2+</sup>, the relative concentration of the two Cu ions is determined from the relative areas under these peaks and the satellite peaks. The Cu<sup>2+</sup> content is found to be more than 70 % for the glasses with x = 0.3, 0.4 while only 44 % for the glass with x = 0.5. The O 1s spectra for all glasses show slight asymmetry and were, therefore, fitted with two contributions, one from bridging oxygen (BO) and the other from non-bridging oxygen (NBO). The ratio NBO/Total oxygen was evaluated for each glass sample and was found to be in good agreement with the theoretical values obtained assuming that CuO behaves as a modifier in these glasses.

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## استخدام مطيافية الإلكترون لدراسة مركبات نحاس التلوريوم الزجاجية

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المستخلص. تم تحضير مواد زجاجية ذات التركيب الكيميائي  $x\text{CuO}-(1-x)\text{TeO}_2$  حيث أن قيم  $x = 0.30, 0.40, 0.50$  وتم دراسة هذه العينات بواسطة مطيافية أشعة إكس (XPS). لقد تم قياس الأطياف الناتجة من عنصر النحاس Cu 2p وعنصر التلوريوم Te 3d وعنصر الأكسجين O 1s. ظهور إشارة "satellite" في طيف Cu 2p دليل على وجود أيونات  $\text{Cu}^{2+}$  في هذه العينات وكذلك عدم وجود تناظر وسع إشارة Cu 2p يدل على وجود أيونات كل من  $\text{Cu}^{2+}$  و  $\text{Cu}^+$  في هذه المواد. تم مقارنة (fitting) إشارة Cu 2p<sub>3/2</sub> بقمي فرق طاقة الربط بينهما 2 eV، والقمة ذات طاقة الربط الأقل ترافقاً  $\text{Cu}^-$  بينما القمة ذات طاقة الربط الأكبر ترافقاً  $\text{Cu}^{2+}$ . تم تحديد نسبة كمية  $\text{Cu}^{2+}$  عن طريق حساب مساحة القمم لإشارة  $\text{Cu}^+$  و  $\text{Cu}^{2+}$  وكانت نسبة  $\text{Cu}^{2+}/\text{Cu}$  تتراوح بين 40% عندما  $x=0.50$  و 90% عندما  $x=0.30$ .

كان خط طيف الأكسجين 1s (O 1s) غير متناظر في كل العينات ولذلك تم مقارنة الإشارة بمساهمتين، الأولى من ذرات الأكسجين المقنطر (BO) والثانية من الأكسجين غير المقنطر (NBO). الأكسجين المقنطر ناتج عن ذرات الأكسجين الموجودة في التركيب Te-O-Te والأكسجين غير المقنطر ناتج عن ذرات الأكسجين الموجودة في التركيب Cu-O-Te. تم تحديد نسبة الأكسجين غير المقنطر في العينات ووجد أنه متناسب مع الكمية المحسوبة نظرياً.

وجد أن طيف إشارة Te 2d متناظرة مما يدل على أن المحيط الكيميائي حول ذرات التلوريوم لم يتغير مع تغير قيم  $x$ .