

## Applications Note

# Smart phone screen – depth analysis of alkali and alkali earth metals

### Overview

In this study we apply the technique of depth profiling to analyse the concentration of elements in the near surface region of smart-phone display glass. We will look at the distribution of alkali and alkali earth metals - in particular K which is a key element in the glass toughening process. Two different depth profiling methods were used, monatomic Ar and Ar cluster, and a comparison is made regarding the ion bombardment effects of these two methods. We emphasise the importance of ion choice when depth profiling inorganic materials in particular those containing light alkali metals.

### Introduction

Soda-lime glass has traditionally been used in automotive and architectural windows however in recent times it has also been employed in smart displays and photovoltaics. Due to this shift in application it has become increasingly important to understand the processes taking place in the surface of the glass for quality control and failure analysis. For electrical devices the mobility and distribution of alkali and alkali-earth metals is particularly important as they can have a pronounced effect on the glass's electrical properties.

Soda-lime glass usually consists of Si, O, Na, Mg, and Ca however K is also used in smart phone displays to reduce the likelihood of fracture under impact. The manufacturing process involves ion-exchange whereby K replaces Na in the surface of the glass – the larger K ions occupy more space in the glass structure creating residual compressive strength – see figure 1.

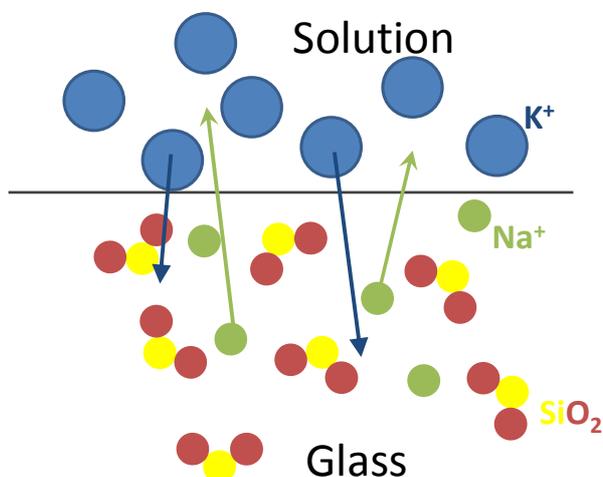


Figure 1. Ion-exchange mechanism

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Here we wish to gain a greater understanding of the metal distribution in toughened glass. We will use depth profiling to analyse the elemental distribution of elements in the surface region of a material. Traditionally this process uses monatomic Ar<sup>+</sup> as the impinging ion however we will discuss the merits of instead using large Argon cluster ions.

### Experimental

All measurements were acquired using a Kratos AXIS Nova photoelectron spectrometer incorporating several features which make high resolution spectroscopic analysis of these types of challenging samples routine:

- A 500 mm Rowland circle Al X-ray monochromator;
- Coaxial charge neutralisation system;
- Magnetic lens;
- 165 mm hemispherical analyser;
- Delay line detector.

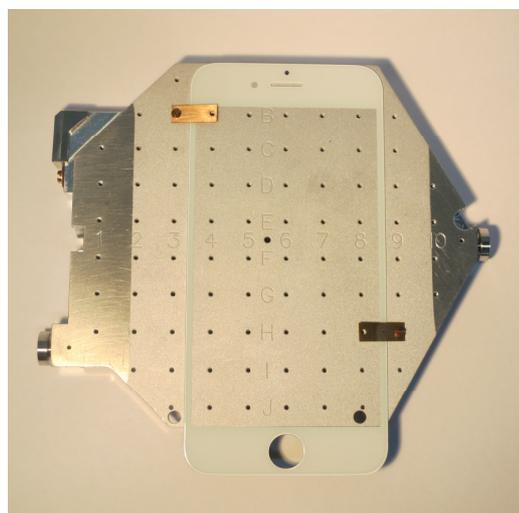


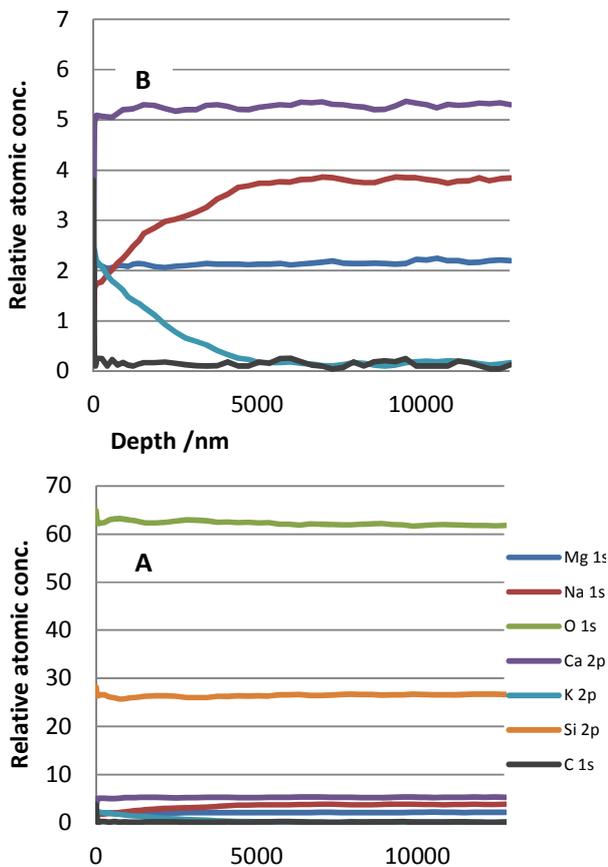
Figure 2: Smartphone screen loaded on sample platen

Due to the large surface area of the sample platen it was possible to load the entire smartphone screen without the need for cleavage and possible loss in integrity. The smartphone screen was mounted on the platen, anchored by two copper clips (figure 2).

Depth profiling was performed using the new Gas Cluster Ion Source. Two different ions were used 5 kV Ar<sup>+</sup> and 20 kV Ar<sub>500</sub><sup>+</sup>. The crater size was 1 mm x 1mm for both profiles with an analysis area of 220µm diameter in the centre of each crater. The etch rate of 20 kV Ar<sub>500</sub><sup>+</sup> was determined as 13 nm/min using a SiO<sub>2</sub> standard.

## Results and Discussion

The XPS depth profile using 5 kV Ar<sup>+</sup> is shown in figure 3a.



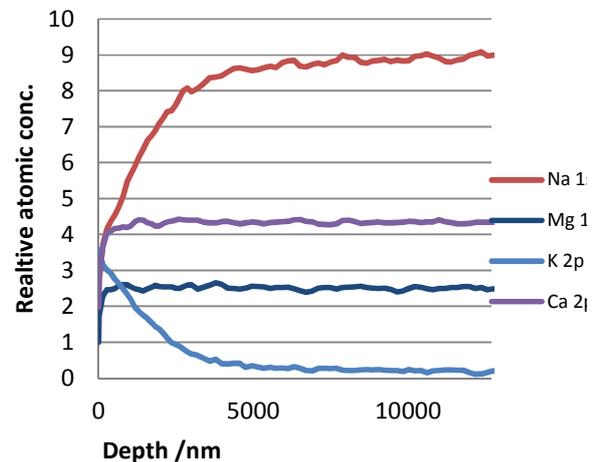
**Figure 3. 5kV monatomic depth profile**

As expected the primary components are Si and O. The low concentration elements are enlarged in Figure 3b. K is highest in concentration (~2.4%) at the surface of the display decreasing to <0.5% at depths greater than 5 µm. Concurrent to the decrease in K is an increase in Na concentration. Interestingly, there is little variation in the other elements present in the surface region remain constant - this highlights the relationship between Na and K as the principle participants in the ion-exchange mechanism. Once the outer K-treated

layer is removed a steady-state Na concentration is reached (< 4%). This value is significantly lower than the expected stoichiometric ration for the soda-lime glass of ~9%.

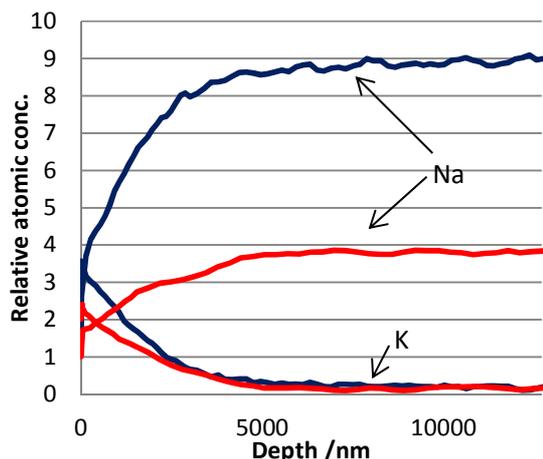
Previous studies have reported decreased Na<sup>+</sup> ions in the surface region under monatomic Ar<sup>+</sup> bombardment<sup>2,3</sup>. The build-up of positive charge in the near surface region repels the highly mobile Na<sup>+</sup> ions into the bulk of the glass. As an ion approaches the glass surface, electron transfer from the surface creates a positive surface layer on the glass. This positive region repels Na ions into the glass matrix creating a sodium-depleted zone. This process is commonly referred to as field-induced migration and leads to an underestimation in alkali element concentration.

For comparison a depth profile was performed using 20 kV Ar<sub>500</sub><sup>+</sup> cluster ions. Once again the highest concentration of K was found at the surface (3.6%) – 33% more than was seen in the monatomic Ar<sup>+</sup> depth profile.



**Figure 4. 20 kV Ar<sub>500</sub><sup>+</sup> depth profile.**

Most significant is the difference in bulk Na concentration using Ar<sub>500</sub><sup>+</sup>, 8.8% compared to <4% using monatomic Ar<sup>+</sup>. This value is also similar to the expected stoichiometric concentration implying negligible field-induced migration occurring under cluster bombardment. This reduction in migration can be attributed to the difference in ion current between the depth profiling methods. Under monatomic Ar<sup>+</sup> the surface experiences an ion current of several µA however in cluster mode the current is less typically 100 times less. This reduction in ion current results in less charge build-up and consequently decreased migration. A comparison of the two modes for Na and K is shown in figure 5 below.



**Figure 5. Comparison of K and Na profiles for monatomic (red) and cluster (blue) depth profiles.**

## Conclusions

Two different depth profiling methods have been applied to a smart phone display to examine metal distribution in the top surface.

Of particular interest was the distribution of K in the surface as this component acts as the toughening agent. K was shown to be present however the concentration decreased to <0.5% once 5 $\mu$ m deep indicating shallow penetration of K in the ion-exchange method.

There was a discrepancy in surface concentration of K for the two modes with the cluster profile showing significantly more surface K. This result, coupled with monatomic Ar<sup>+</sup> underestimating bulk Na concentration, highlights the limitations of monatomic depth profiling due to unwanted effects of field-induced migration of Alkali metal. The effect is not as pronounced as found with Na due to ion mobility decreasing with increasing ionic radii.

These results indicate XPS combined with Ar cluster ion sputtering as a simple, reliable depth analysis method for alkali glass.

## References

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