Chalcogenide Advanced Manufacturing Partnership Open Day

Chalcogenide Glass Corrosion Studies

Emma Metcalfe-Smith

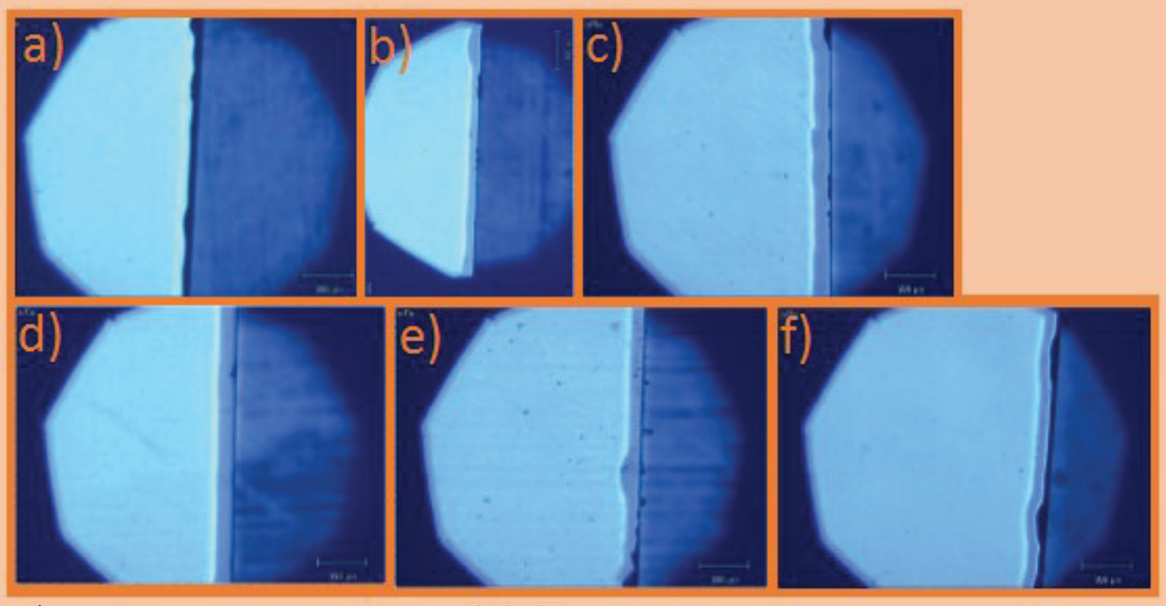
Optoelectronics Research Centre – University of Southampton, Southampton, SO17-1BJ, UK

INTRODUCTION

GLS, Gallium Lanthanum Sulphide, is a non-toxic semiconducting chalcogenide glass, transparent in the infrared, providing a wide variety of applications. To confirm its versatility, chemical resistivity tests and an investigation into the effects of various solvents upon GLS samples were conducted.

THE EFFECTS OF GLS EXPOSED TO SOLVENTS FOR 2 WEEKS

GLS slides, 10mm x 10mm x 1mm in dimension, were exposed to 20ml of a particular solvent and left for a period of two weeks. After exposure, for which no visible changes to the naked eye were seen, the slides were washed down with methanol and blasted with nitrogen gas. Subsequent photographs were then taken with an optical microscope to seen the effects upon the GLS surface:



↑ *Images taken with optical microscope (5x). a) Control sample, no solvent exposure. Other GLS slides exposed to b) Acetone, c) Methanol, d) Propylamine, e) Butylamine, f)Propan-2-ol*

No visible degradation could be seen.

ALKALI AND PHOSPHATE RESISTANCE TESTS

The alkali (AR)[1] and phosphate resistance (PR)[2] classes are International Standard tests intended to indicate the resistance of optical glasses to aqueous alkaline solutions in excess. These are of particular interest as warm alkaline solutions are routinely used in the cleaning processes for polished surfaces. The phosphate resistance takes into consideration any detergents that aren't pure hydroxide solutions.

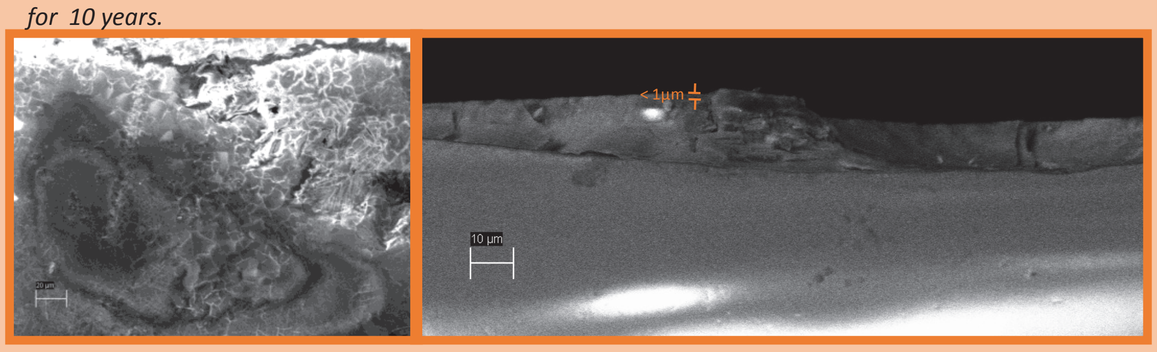
AR is conducted with sodium hydroxide, c=0.01 mol/l, pH=10, and PR with pentasodium -triphosphate, c=0.01 mol/l, pH=10. Both tests are based on the time required to remove a layer thickness of glass of 0.1µm at 50°C. In both instances , the degradation is minimal and does not exceed 0.1µm after an exposure time of 4 hours. A highest classification of grade 1 is awarded.

	Alkali Resistance-Sodium Hydroxide		
Time	**Initial Weight (g)**	**Final Weight (g)**	**layer thickness degraded (µm)**
15 mins	0.422607	0.422583	0.059
1hr	0.421307	0.421282	0.061
4hr	0.424877	0.424843	0.083
	Phosphate Resistance-Pentasodiumtriphosphate		
Time	**Initial Weight (g)**	**Final Weight (g)**	**layer thickness degraded (µm)**
15 mins	0.423867	0.423842	0.061
1hr	0.421343	0.421314	0.072
4hr	0.422933	0.422895	0.095

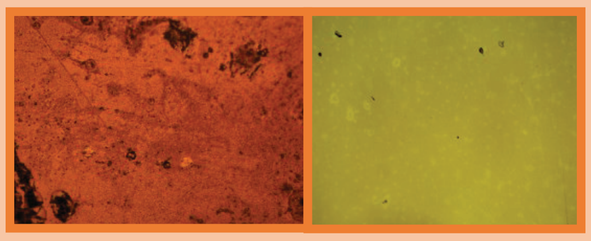
THE EFFECTS OF GLS EXPOSED TO WATER FOR 10 YEARS

↑ *GLS sample exposed to water for 10 years.*

In June 2004, a GLS sample was sealed in deionised water. In February 2015 this sample was recovered and the long term effects were analysed. The stagnant water was recorded to have a pH of 8.05 (typical deionised water pH=7.51). As can be seen by the SEM images, the water had ingressed up to a maximum depth of 1µm into the sample.



SEM images of GLS sample. Left) Surface Image of GLS, the top right area shows significant mould growth, the dark pools on the bottom left indicate the corrosion caused to the surface. Right) A small fragment chipped off allowing a scan to determine the water ingression boundary from the surface.

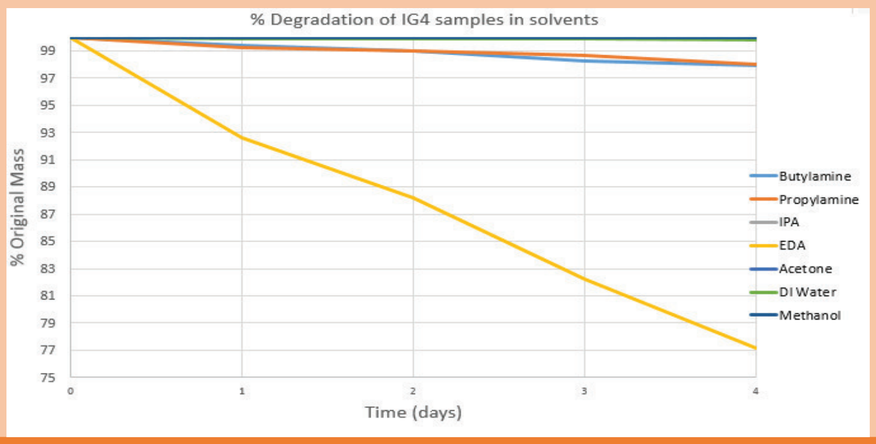


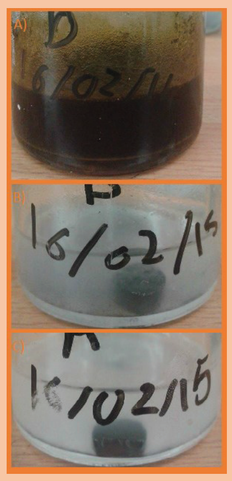
↑ *Optical microscope (x20) Left) GLS sample exposed to water for 10 years. Right) 10 year old control sample*

The sample was heated and cleaned, to remove the mould spores that had grown on the GLS. Photographs were then taken with the optical microscope. As can be seen there is moderate corrosion upon the surface of the sample but this is superficial given the SEM analysis.

COMPARING GLS TO IG4

Made of the components Ge-As-Se, IG4 is a leading chalcogenide with exceptional transmissions. However, it's poor chemical resistivity is apparent when its exposure to solvents is compared to that of GLS. (the samples exposed to butylamine and propylamine respectively lost over 2% of their initial mass and the sample exposed to EDA lost 23% in just 4 days.)





↑Above, IG4 exposed to A) EDA, B) Propylamine, C) Butylamine after 4 days

←% degradation of IG4 for various solvents as a function of time

CONCLUSION

These glass corrosions studies have confirmed the excellent chemical resistivity of GLS when exposed to a multitude of solvents over a short time frame. Surface damage was seen on the sample exposed to water for 10 years, and the moisture identified within the sample was nominal.

[1] ISO 10629: Raw optical glass - Resistance to attack by aqueous alkaline solution at 50°C - Test method and classification; July 1996

[2] ISO 9689: Raw optical glass-Resistance to attack by aqueous alkaline phosphatecontaining detergent solutions at 50°C-Testing and classification; December 1990

Grant Reference: EP/H02607X/1 - EPSRC Centre for Innovative Manufacturing

EPSRC Pioneering research and skills

Research Sponsored by EPSRC, the Engineering & Physical Sciences Research Council

ChAMP Open Day is hosted by: **Optoelectronics Research Centre**
Event administration by **Xmark Media**.