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सारांश

अनेक सेंटीमीटर मोटाई एवं बृहत् विमा युक्त उच्च घनत्व वाले सीसा कांचों का हॉट सेल में परिरक्षण गवाक्ष में प्रयोग किया जाता है । दृष्यता को बढ़ाने के लिए आप्टिकली पॉलिश की गई सतहों से प्रकाश के परावर्तन का न्यूनीकरण करना पड़ता है ताकि प्रसारण को बढ़ाया जा सके क्योंकि मोटे कांच में प्रकाश के अवशोषण को रोका नहीं जा सकता । इस प्रयोजन के लिए निम्न रिफ्रैक्टिव इंडेक्स वाले पदार्थ के परावर्तनरोधी कोटिंग की आवश्यकता है ।

ग्लैशल ऐसिटिक अम्ल में परिवेश तापमान पर सीसे के वरणात्मक निक्षालन से कांच की सतह पर सिलिका समृद्ध निक्षालित परत का विकास होता है । चूंकि सिलिका की रिफ्रैक्टिव इंडेक्स कम होती है, निक्षालित परत परावर्तनरोधी कोटिंग का काम करती है ।

दो आप्टिकली पॉलिश किये गये परिरक्षण गवाक्ष के कांचों को ग्लैशल ऐसिटिक अम्ल में 2, 5 और 10 दिनों के लिए परिवेश तापमान पर निक्षालित किया गया एवं निक्षालन प्रभाव को देखने हेतु उनके परावर्तन एवं प्रसारण स्पेक्ट्रा निकाले गये थे । पारदर्शी कांचों के लिए प्रसारण को 10 दिनों के निक्षालन के पश्चात 78.76%से 85.31% तक बढ़ाया जा सका । कांच से परावर्तन को 12.48 से 11.67% तक घटाया जा सका । रंजित कांचों के लिए 5 दिनों के निक्षालन के पश्चात प्रसारण 87.77% से 88.24% तक बढ़ाया जा सका जबकि इसी अवधि के दौरान परावर्तन 12.28% से 5.6% तक कम हुआ । प्राप्त डाटा के आधार पर परावर्तनरोधी लेपन के विकास हेतु 10 दिनों तक एवं रंजित परिरक्षण गवाक्ष कांचों के लिए 5 दिनों तक निक्षालन की सिफारिश की गई है ।

इस प्रक्रिया को ऐसिटिक अम्ल से भरे किसी विमा की कुछ उच्च डाइमेन्शन वाली पीवीसी टंकी का संविरचन कर परिरक्षण गवाक्षों के लिए प्रयोग किया जा सकता है ।

आधारभूत शब्द

परिरक्षण गवाक्ष कांच
परावर्तनरोधी लेपन
ऐसिटिक अम्ल
परिवेश तापमान

एनके/एसडी 12.12.05

Abstract

High density lead glasses having thickness of several centimetres and large dimensions are used as shielding windows in hot cells. To improve visibility, the reflection of light from its optically polished surfaces needs to be minimized to improve transmission as absorption of light in the thick glasses can not be avoided. Antireflectance coating of a material having low refractive index is required for this purpose.

Selective leaching of lead at ambient temperature in glacial acetic acid develops a silica rich leached layer on glass surface. Since silica has low refractive index, the leached layer serves as antireflectance coating.

Two optically polished discs of shielding window glasses were leached in glacial acetic acid at ambient temperature for 2, 5 and 10 days and their reflectance and transmittance spectra were taken to find effect of leaching. For transparent glass transmittance could be improved from 78.76 % to 85.31 % after 10 days leaching. Reflectance from the glass could be decreased from 12.48 to 11.67 %. For coloured glass transmittance improved from 87.77 % to 88.24 % after 5 days leaching while reflectance decreased from 12.28 % to 5.6 % during same period. Based on data generated, 10 days leaching time is recommended for developing antireflectance coating on transparent shielding window glass and 5 days for coloured shielding window glass.

The procedure can be used for shielding windows of any dimensions by fabricating a PVC tank of slightly high dimensions and filling with acetic acid.

Keywords

Shielding window glass
Antireflectance coating
Acetic acid
Ambient temperature

Contents

1. INTRODUCTION

Hot cells designed to handle high gamma radiation have [1]walls measuring a few meters in width. To observe the operations in side the hot cell, windows are provided. These are fitted with special glasses capable of absorb high energy radiation and having transparency to visible light. The glasses are several inches thick to provide good shielding from gamma radiations to the operators.

The light incident on a surface is reflected, transmitted or absorbed in the medium. According to Beer-Lambarts law, the light absorption by the medium is proportional to the path length. Since absorption of light by the shielding window glasses can not be avoided, its transmission can be improved by decreasing reflectance from the surface. This is achieved [2,3] by applying antireflectance coating to the glass surfaces.

Most of the techniques to apply antireflectance coatings are patented [4 to 7] and very little information is available in abstracts. At the same time, special techniques are required for applying antireflectance coatings on shielding window glasses due to their high thickness and large dimensions. Hence work was undertaken based on literature information to standardize the reflectance coating procedure which can be used on actual shielding windows of practically any dimension. The report discusses the results of the technique developed for the above purpose.

2. ANTIREFLECTANCE COATING METHODOLOGY

2.1 Need for Antireflectance Coating

When pencil of light impinges on a glass surface, a large part of it is transmitted after refraction. A small part , approximately 6 to 8 % is reflected[2]. The reflected light decreases contrast of image or even produces bright spot at its centre. For thick glasses like shielding windows, due to partial absorption of light by glass, reflectance should be minimized to enhance transmittance. Modification of this sort is made by depositing one or more thin films/layers of various materials. In general this stack of layers is referred to as a stratified medium.

To minimize reflectance, the refractive index n_f this film should be [8] geometric mean of indices n_1 and n_2 of incident and refracting media-

$$n_f^2 = n_1 * n_2$$

and film thickness d_f should be equal to a quarter of wave length of light in the film material i.e. it should satisfy the equation-

$$n_f d_f = \frac{\text{wave length}}{4}$$

where wave length is vacuum wave length of light for which reflectance of the surface is to be minimised.

Such film is called a quarter layer. This causes a phase shift of half wave length of the incident light leading to its extinction of reflected light which occurs under these conditions. From this, it is apparent that total extinction can be achieved at one particular wave length for particular thickness of coating of a antireflectance material. In general this lies at about 550 nm and residual reflectance assumes a purple tinge. Multiple coating treatment makes it possible to achieve practically complete extinction over whole visible region of light spectrum.

2.2 Coating Materials

The refractive indices of glasses are generally near 1.5. The refractive index of a glass depends on its chemical composition. Presence of heavy elements like barium and lead increases [9] the refractive indices upto 1.7 depending upon their content. Magnesium fluoride and silica have refractive indices of 1.38 and 1.45 respectively. Oxides of zirconium, yttrium and titanium have refractive indices of 1.96, 1.74 and 2.11 respectively. Hence MgF_2, and silica are most preferred for antireflectance coatings on glasses. Using MgF_2, reflectance of glass surface can be reduced[8] to 0.5 %. If second and third layer of coating is desired, ZrO_2, Y_2O_3 and TiO_2 are used.

2.3 Coating Procedures

MgF_2 is manufactured[10] by adding HF to cationic resins in Mg(II) form. It can be applied onto glass surfaces by chemical vapour deposition techniques. For this glass substrates are required to be heated to 480 to 650 °C. This technique is too complex for coating shielding window glasses due to their very high thickness. Glasses are poor conductors of heat, hence the chances of developing cracks or crevices due to uneven heating are large. Other technique of coating glass surface with MgF_2 described in literature is [11] by applying magnesium salts like $MgSO_4$ and NH_4F and heating. This technique also requires heating of glass surface and not applicable for shielding windows.

Silica can not be deposited on glass surfaces due to its extremely high boiling point and poor bonding it can achieve with glass surface by chemical reactions. The fact that silica is one of the glass forming component can however be used to create a silica layer in situ on glass surface. Here we take the advantage of leachability of glasses in water or other corrosive media. Nuclear waste glasses and borosilicate glasses are known[12,13] to leach incongruently. Generally the counter ions like those of alkali, alkaline earth and lead leach more selectively leaving behind leached layer of higher silica content on the glass surface.

We have taken advantage of this fact. Unlike alkali metal oxides, the lead oxide is only sparingly soluble in water. Hence a more aggressive leachant is required than water used for nuclear waste glasses. Lead Oxide reacts with acetic acid forming sparingly soluble lead acetate. Lead acetate has[14] solubility of 0.85, 1.15 and 2.36 gm/100 g of water at 20, 30 and 60°C respectively. Hence acetic acid was selected as leachant.

Literature suggests[7] use of 60°C as leaching temperature. Since acetic acid is highly corrosive and having boiling point of 117°C, electrical heaters, leaching tanks to accommodate large sized shielding windows need to be fabricated from special materials. Acetic acid is highly corrosive to stainless steel, and deposition of iron, chromium and nickel compounds on shielding window surfaces is likely. Also 60° C temperature will release significant quantities of acetic acid vapours. The leached layer formation increases with square root of time. Hence we decided to continue leaching at ambient temperature for longer time instead of using elevated temperatures and shorter time durations. The leaching tanks can be fabricated PVC around the shielding window blocks of any dimensions for this purpose to handle acetic acid. LR grade glacial (17M) acetic acid was used as such with out dilution.

3. EXPERIMENTAL

Shielding window glass discs having 15 mm diameter and 6.3 mm thickness were supplied by Central Glass and Ceramic research Institute, Kalkata. They were already optically polished on both sides of circular surface. The transparent and coloured glasses had specific gravities of 3.85 and 5.2 respectively.

Transmittance and reflectance spectra in ultraviolet and visible regions were taken using spectrophotometer having such facility before start of leaching.

The glass discs were tied on the cylindrical surface by a rolled Teflon tape and then suspended inside a Teflon beaker filled with LR grade glacial acetic acid. They were removed after 2 days, washed with demineralised water and dried under IR lamp. The leached discs were subjected to reflectance and transmittance specta as earlier.

Leaching was continued for additional 3 and 5 days, each time subjecting them to spectral analyses before further leaching. In all these operations, the discs were handled without touching the optically polished surface.

The results of reflectance and transmittance spectra for transparent glass are presented in Fig 1 and 2 respectively. The results of reflectance and transmittance spectra for coloured glass are presented in Fig 3 and 4 respectively .

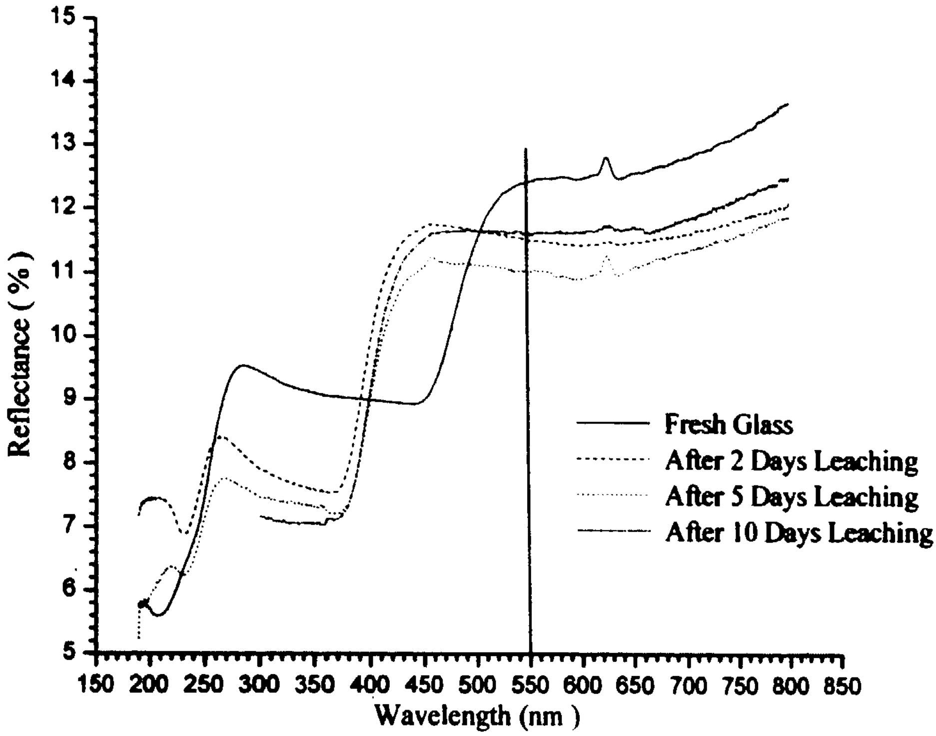

Fig 1. Effect of leaching on light reflectance
on transparent shielding window glass

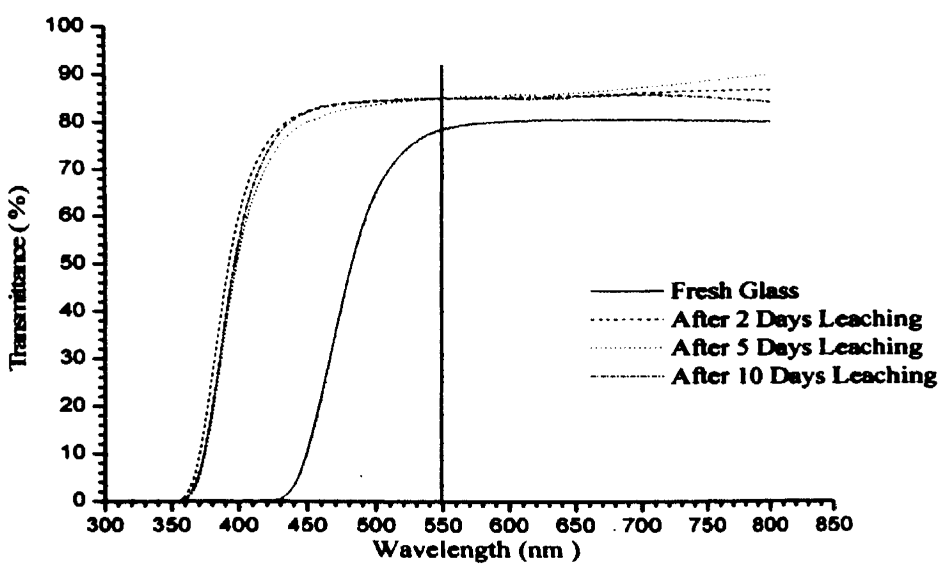

Fig 2. Effect of leaching on light transmittance
on transparent shielding window glass

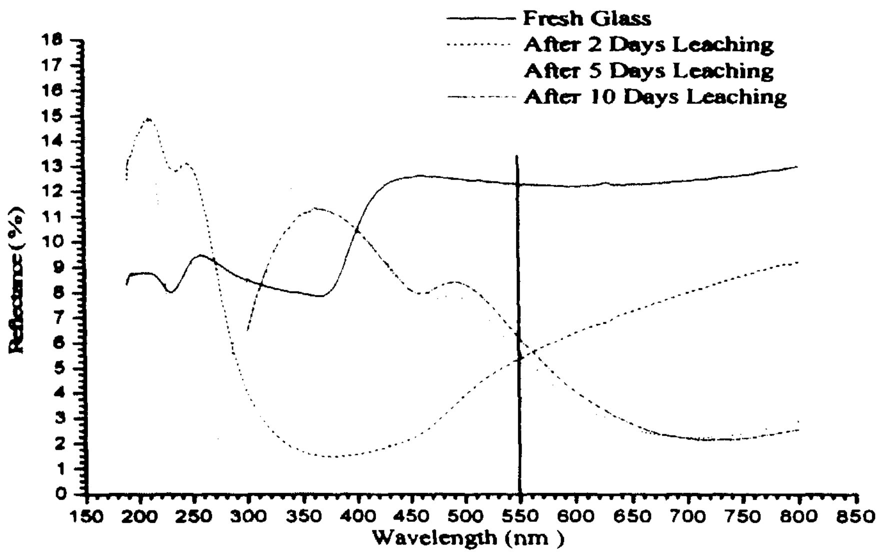

Fig 3. Effect of leaching on light reflectance
on coloured shielding window glass

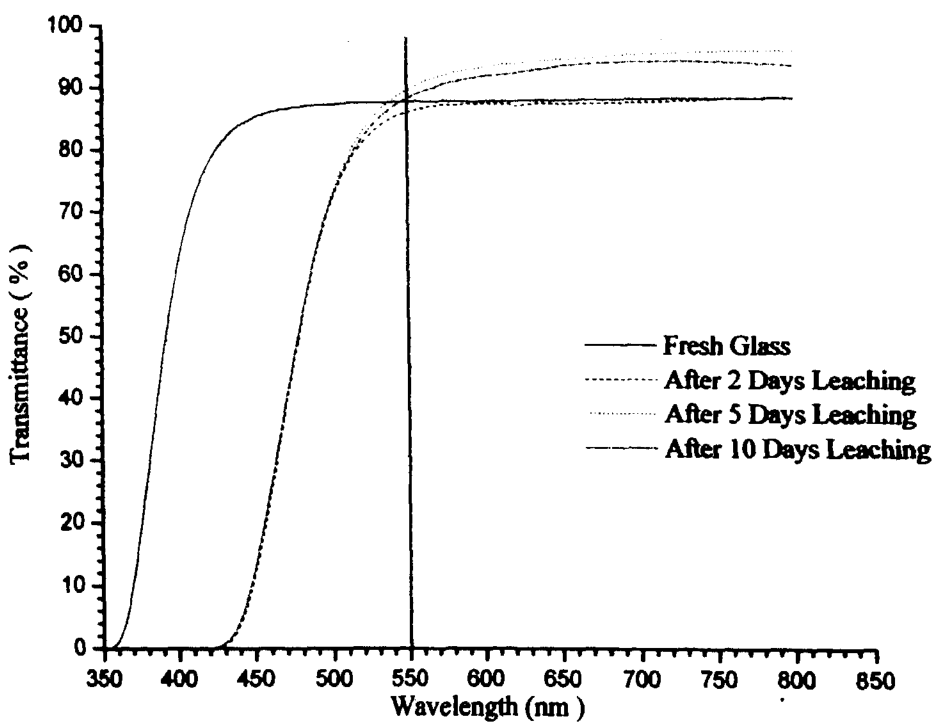

Fig 4. Effect of leaching on light transmittance
on coloured shielding window glass

4. RESULTS AND DISCUSSIONS

As Indicated in 2.1, the elimination of reflectance can be achieved at the coating thickness equal to a quarter of wavelength of interest. Hence effectiveness of a antireflectance coating is depended on wavelength of light. For practical purpose the values at 550nm are reported in literature. However entire visible region of light spectrum from 400 to 760 nm should also be considered for selecting antireflectance coating. The application of antireflectance coating should not lead to increased absorption of light. Hence both reflectance and transmittance spectra are to be considered for deciding type and thickness of antireflectance coating.

As seen in Fig. 1, the reflectance on transparent glass surface at 550 nm decreased from 12.48 % to 11.07 % in 5 days of leaching but again increased to 11.67 % for 10 days leaching. At wave lengths longer than 550 nm, the decrease in reflectance was more as compared to that at wavelengths shorter than 550 nm. Thus the leached layer was more effective in red region. At 400 to 500 nm the leached layer in fact increased the reflectance of light from transparent glass.

As seen in Fig. 2, there was a marked improvement in transmittance of light after formation of leached layer coating on the transparent glass. Appreciable improvement in light transmittance was observed in the region from 360 to 430 nm. The transmittance of light had improved through out the visible region of light spectrum as leaching period progressed. At 550 nm the transmittance improved from 78.76 % to 85.1 % after 2 days of leaching and 85.31 % after 10 days of leaching.

Thus, the reflectance of transparent glass decreased by about one percent but transmittance improved by 7 percent after leaching in acetic acid.

Hence for transparent glass, leaching of optically polished surface for 10 days in glacial acetic acid is optimum to develop good quality antireflectance coating.

As seen in Fig. 3, for coloured glass, the reflectance at 550 nm decreased from 12. 28 % to 5.38 % after 2 days of leaching and then rose slowly to 5.6 and 6.1 % with increasing leaching period to 5 and 10 days respectively. For all 3 leaching periods the reflectance remained in narrow range of 5 to 6 % for light of 540 to 570 nm. As seen in Fig. 3 , the 2 days leaching decreased reflectance in the visible region of 400 to 550 nm. Leaching for 5 and 10 days proved to be more effective in decreasing reflectance in vision region from 550 to 760 nm. Hence 3 to 4 days leaching period seems to be optimum to decrease reflectance throughout the visible region of light spectrum.

From Fig. 4, it is seen that the transmittance at the 350 to 530 nm wave length decreased significantly for the coloured glass. This trend is reverse as that observed for transparent glass. For visible region of spectrum from 400 to 430, the transmittance was practically absent. At 550 nm, the transmittance initially decreased from 87.77 % to 86.08 % for 2 days leaching period. The transmittance subsequently improved to 89.62 % and 88.24 % with increased leaching periods of 5 and 10 days.

Thus for coloured glass, leaching period of 4 to 5 days is considered to be optimum to minimise reflectance and improve transmittance. For coloured glass, the reflectance decreased by about 6 % but transmittance improved by only one percent. This is due to absorption of light by leached layer.

As the reflectance of both types of glasses remained above 5 percent, there is enough scope for improving their antireflectance properties . The leached layer thickness and its refractive index are critical for selection of leaching period. As the composition and depth of leached layer vary continuously with time of leaching, a profile of leached layer can be very useful to optimise leaching conditions like time and acetic acid concentration. In this work we used 6.3 mm thick optically polished samples. Due to high sample thickness, depth profile of leached layer could not be carried out using Rutherford Back Scattering Spectroscopy. Use of such techniques are surely useful to fine tune the leaching procedure to decrease reflectance.

5. CONCLUSIONS

Silica leached layer developed on shielding window glasses by selective leaching of lead can serve as anti reflectance coating. For transparent glass having specific gravity of 3.85, transmittance could be improved from 78.76 % to 85.31 5% after 10 days leaching in glacial acetic acid. Reflectance from the glass could be decreased from 12.48 % to 11.67 % during same period For coloured glass having specific gravity of 5.2 , transmittance improved from 87.77 % to 88.24 % after 5 days leaching while reflectance decreased from 12.28 % to 5.6 % during same period. Based on data generated, 10 days leaching time is recommended for developing antireflectance coating on transparent shielding window glass and 5 days for coloured shielding window glass. The technique can be standardized for glass on any composition to obtain most effective coating.

The proposed antireflectance coating method can be fine tuned by analyzing depth profile of leached layer by Rutherford back scattering spectroscopy using discs of 1 to 2 mm thickness. The proposed treatment method works at ambient temperature and can be deployed to shielding windows of any dimensions.

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