NEW GENERATION PROCESSING OF GLASS BY CHEMICAL TEMPERING

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Master's Thesis

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Programme in Eskisehir Technical University, Graduate School of Sciences

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Eskişehir

Eskişehir Technical University

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ABSTRACT

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Chemical tempering is one of the important methods used to increase strength of glass. It is widely used in industry like aircraft and display devices. A novel method of chemical tempering was conducted using ordinary furnace to achieve a temperature of 450, 500, and 550 ^{o}C, for several hours. A clay coating paste was used on glass. This clay mixed with potassium nitrate salt with two different volume concentrations (46.7 vol.%, 63.7 vol.%). Three contact time (8, 12, 16 hours) were used in this study. Also, two different glass slide thicknesses (1 mm, 4mm) were used. XRF test used to determine chemical composition of glass, surface residual stresses test to determine optical properties, hardness test (H_V), (3-points) bending test, SEM, EDX to determine the depth of potassium penetration, FTIR test to measure the transmittance of light and ballistic impact test to determine strength of glass for bulletproof applications. The achieved results were shown a quality similar to glass tempered with the method using molten salt bath Furance. The strength of glass was increased about 3.5 folds in case of thin glass, 3 folds increased in case of thick glass tempered at 450 ^{o}C, and increased 4.75 folds in case of thick glass tempered at 500 ^{o}C, compared with untreated glass. While optical and mechanical properties were failed for the thick glass tempered at 550 ^{o}C. The hardness was increased about (15 % in case of thin glass), and in case of thick glass (9 % for glass tempered at 450 ^{o}C, 14% for glass tempered at 500 ^{o}C, and 12% for glass tempered at 550 ^{o}C). The depth of potassium penetration was 20 μm in case of thin glass, and 15,30,70 μm for 450,500,550 ^{o}C respectively for tempering process of thick glass. The transmittance of glass increased about 12% in case of thin tempered glass, and decreased about 13% for thick glasses tempered at 500, 550 ^{o}C, in addition to that, the thick glass tempered at 450 ^{o}C did not show any change in transmittance.

Keywords: Chemical tempering of glass, strengthened glass, Toughened glass

ÖZET

CAMLARINI YENİ NESİL KİMYASAL TEMPERLEME SÜRECİ

Alaa Badr Hasan HASAN

Malzeme Bilimi ve Mühendisliği Anabilim Dalı

Eskişehir Teknik Üniversitesi, Lisansüstü Eğitim Enstitüsü Bilim Dalı

Eskişehir Teknik Üniversitesi, Lisansüstü Eğitim Enstitüsü, Temmuz 2020

Danışman: Dr. Öğr. Üyesi Emrah DÖLEKÇEKİÇ

Kimyasal temperleme, camın mukavemetini artırmak için kullanılan önemli yöntemlerden biridir. Hava taşıtları ve görüntü aygıtları gibi sektörlerde yaygın olarak kullanılmaktadır. Kimyasal temperlemenin yeni bir yöntemi birkaç saat içinde 450, 500 ve 550 ^{o}C sıcaklığa çıkabilen sıradan bir fırın kullanılarak gerçekleştirildi. Cam üzerine bir kil kaplama macunu kullanılmıştır. Bu kil, iki farklı hacim konsantrasyonunda (hacimce % 46.7 ve hacimce % 63.7) potasyum nitrat tuzu ile karıştırıldı. Bu çalışmada farklı üç temas süresi (8, 12, 16 saat) kullanılmıştır. Ayrıca iki farklı kalınlıkta (1mm ve 4mm) cam kullanılmıştır. Camın kimyasal bileşimini belirlemek için XRD testi, optik özellikleri belirlemek için yüzey kalıntı gerilme testi, sertlik testi (H_V), 3 noktalı eğme testi, SEM, potasyum penetrasyon derinliğini belirlemek için EDX, ışık geçirgenliği ölçümü için FTIR testi ve kurşun geçirmez uygulamalarda camın dayanımını belirleme için balistik çarpışma testi kullanıldı. Elde edilen sonuçlar, erimiş tuz banyosu fırını metoduyla temperlenmiş cam ile benzer kalitede olduğu göstermektedir. İnce cam durumunda camın mukavemeti yaklaşık 3.5 kat, işlem görmemiş cama göre 450 ^{o}C'de temperlenmiş kalın cam durumunda 3 kat artmış ve 500^{o}C'de temperlenmiş kalın cam durumunda 4.75 kat artmıştır. 550 °C'de temperlenmiş kalın camda optik ve mekanik özellikler başarısız olmuştur. Sertlik ince camda yaklaşık %15 ve kalın camda 450^{o}C de cam için % 9, 500 ^{o}C de temperlenmiş cam için %14 ve 550 ^{o}C'de temperlenmiş cam için %12 artmıştır. İnce cam durumunda penetrasyon derinliği 20 μm ve 450, 500 ve 550 ^{o}C'de temperlenmiş ince camda sırasıyla 15,30 ve 70 μm idi. İnce temperli cam durumunda camın geçirgenliği yaklaşık %12 arttı, 500 ve 550 ^{o}C'de temperlenmiş kalın camlar için yaklaşık %13 azaldı, 450 ^{o}C'de kalın cam değişim göstermedi.

Anahtar Sözcükler: Kimyasal camın temperlenmesi, Güçlendirilmiş cam, Sertleştirilmiş cam

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Alaa Badr Hasan HASAN

03/07/2020

STATEMENT OF COMPLIANCE WITH ETHICAL PRINCIPLES AND RULES

I hereby truthfully declare that this thesis is an original work prepared by me; that I have behaved in accordance with the scientific ethical principles and rules throughout the stages of preparation, data collection, analysis and presentation of my work; that I have cited the sources of all the data and information that could be obtained within the scope of this study, and included these sources in the references section; and that this study has been scanned for plagiarism with "scientific plagiarism detection program" used by Eskişehir Technical University, and that "it does not have any plagiarism" whatsoever. I also declare that, if a case contrary to my declaration is detected in my work at any time, I hereby express my consent to all the ethical and legal consequences that are involved.

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GLOSSARY OF SYMBOLS AND ABBREVIATIONS

ASTM	:	American society for testing and materials.
BR2	:	level 2.
BR6	:	Level 6.
E	:	Elastic modulus.
EVA	:	Ethelyn-vinyl acetate.
CIRON	:	Cation induced relaxation of network.
CP	:	Complete penetration.
EDX	:	Energy dispersive X-Ray spectroscopy.
FTIR	:	Fourier transform Infrared spectroscopy.
GM	:	Glass matrix.
H_V	:	Vickers hardness.
IS	:	Ion source.
IX	:	Ion exchange.
K_{IC}	:	Critical stress factor.
K_I	:	Opening in tensile stress.
K_{II}	:	Sliding in shear stress.
K_{III}	:	Tearing in shear stress.
MOR	:	Modulus of Rapture.
MPa	:	Mega Pascal.
NP	:	No Penetration.
PC	:	Poly carbonate.
PP	:	Partial Penetration.
PVB	:	Poly vinyl butyral.
r_o	:	Atomic materials component equilibrium distance.
SEM	:	Scanning Electron Microscope.
SIF	:	Stress intensity factor.
σ_{th}	:	Theoretical strength.
U	:	Total energy.
UV	:	Ultraviolet.
XRF	:	X- Ray Fluorescence spectroscopy.
Y_f	:	Geometric factor.

1. INTRODUCTION

1.1. Glass History:

Glass is one of the oldest products going back to 5000 B.C. According to archeological facts, the glass was first made in the east. Mesopotamia and Egypt approximately in 3500 AD. Over the next 500 years, Egypt, Syria among other Countries along the coast of the Mediterranean Sea have become centers for the production of glass. The main method of processing of glass is long and hard due to the use of primitive steel ovens. Glass production was for bottles, container and Cups, and then applied to the house decoration utilizing painted and etched glass. It was a long journey to the glass before it grew to what is seen today. The journey didn't end there. Advanced glassmaking methods are often energy-dependent. Today technologies use electrical furnaces [1].

1.2. Glass in Definition:

In public language the term glass designates a transparent substance, possessing the properties of hardness, rigidity, and brittleness, and, apart from transparency, these are the typical properties one normally associates with a solid. Glass also possesses a number of properties which are characteristic of the liquid state, and classification of glass as a liquid of very high viscosity rather than a solid would be in accordance with modern views. Unlike crystals, glass does not have a sharp melting point, but like crystalline solids, glasses show elasticity. Due to the complexity of the structure of glass, all-encompassing definition for glass remains elusive, and instead a number of definitions have been suggested over the years. A more generally accepted definition is that offered by ASTM (C162) which states “a glass is an inorganic product of fusion which has cooled to a rigid condition without crystallization.” However, the ASTM definition limits the definition of glass to inorganic constituents, which fails to explain organic and molecular glasses that now represent a rapidly growing area of study [2]. Newly developed solution-based sol–gel synthesis of oxide materials occurs at much lower temperature than traditional solid-state fusion processes and also allows powderless non-fusion processing of glasses. X-ray and electronic diffraction studies have shown that glass lacks long-range periodic atomic arrangement, and every type of glass exhibit time-dependent glass transformation behavior [2]. Pointing this out, Varshneya and Mauro suggest a scientific definition of glass as “a solid having a non-crystalline structure, which continuously converts to a liquid upon heating.” Zarzycki favors a more simplified definition: “a glass is a non-crystalline solid exhibiting the phenomenon of glass transition. “Thereby this definition also conveniently separates non-crystalline materials into the categories of glass and amorphous materials [2].

1.3. Structure of Glass:

There are many models that characterize the glass system, including a random network model, a crystallite model and other versions of the system. Random theory of networks focused originally on Zachariasen's ideas is the most common model used for the construction of glass. The following literature contains descriptions of other models [2]. Here, only a random network model is addressed briefly. The random network model portrays glass as a three-dimensional, symmetrical and periodic network where no element is replicated periodically. Such networks compose of oxygen polyhedra for oxide glass. Supposing that the energy content of a glass should be identical to that of the corresponding crystal, Zachariasen [2] suggested four laws on oxide glass forming:

- An oxygen atom should be linked to not more than two glass forming atoms.
- The coordination number of glass-forming atoms is low, 4 or less.
- Oxygen polyhedra share corners, not edges or faces.
- For 3-D networks, at least three corners of each polyhedron should be shared.

Different networks in the glass shape the connecting force that shapes the network framework in crystalline silica (SiO_2) (see Figure 1.1 (a)) and is known as network formers. When alkaline silicates are quickly made up of a glass, the sodium ions are part of the material as alkali oxides such as Na_2O are introduced into silica glass and hold random locations scattered across the surface to ensure neutrality of charge (see. Figure 1.1 (a), (b)).

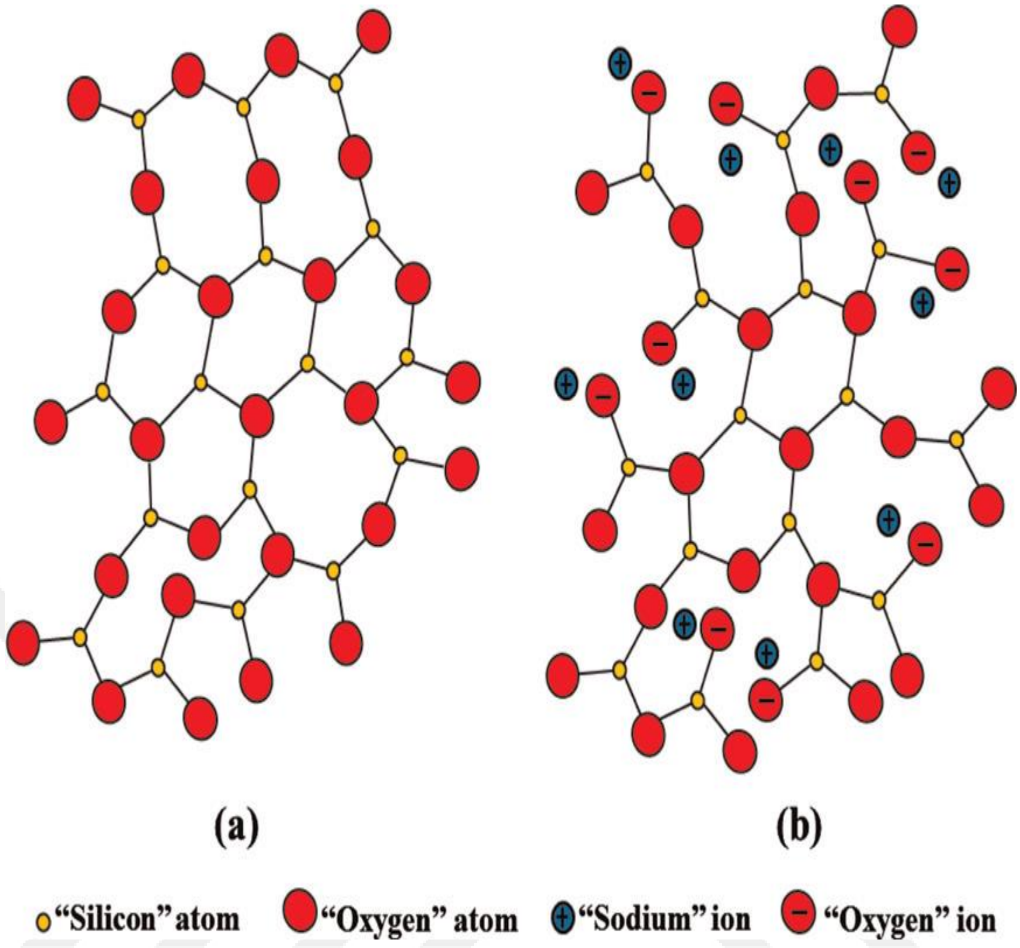

Figure 1.1. *Schematic two-dimensional glass structures :(a) amorphous SiO_2 network; (b) SiO_2 network modified through addition of Na_2O [2].*

These alkali oxides (e.g., Na_2O, K_2O, Li_2O, CaO, MgO, and PbO) provide additional oxygen ions that modify the network structure. For this reason, they are known as network modifiers. With an increase in the number of modifiers, the ratio of oxygen to silicon also increase sand breaks up the three-dimensional network with the formation of singly- bonded oxygens that do not participate in the network, thus reducing the length of the chain. The principal effect of these modifiers is a reduction in melting temperature and working temperature by decreasing the viscosity. An excess of modifiers can make melting sufficiently simple and mobile that crystallization occurs in preference during the formation of glass. Cations of higher valency and lower coordination number than the alkalis and alkali earth metals may act as both network formers and modifiers and are referred to as inter-mediates; for example, ZrO_2, TiO_2, Al_2O_3, ZnO.

1.4. Soda Lime Glass:

Soda-lime glass is one of the most common commercial glass in the world. Soda lime glass also called soda lime silicate (SLS). It is relatively inexpensive and recyclable. A common composition of this glass is 70–75 wt per cent SiO_2, 12–16 wt per cent Na_2O and 10–15 wt. per cent CaO [2]. A lot of other different properties and product criteria require other glass composition. The key component of this kind of glass, other than silica (SiO_2), is sodium oxide or soda (Na_2O). Although sodium oxide contains oxygen atoms, it is still maintained Ionic rather than covalent bonds together. The sodium atoms in the mixture donate the electrons to the oxygen atom, binding of negatively charged oxygen ions and positively charged sodium ions. The oxygen atom with the extra electron attaches to the One silicone atom and does not create a connection between pairs of silicone atoms. So, the melting temperature of the mixture is significantly diminished [2]. Relatively high alkali production of glass often induces a rise of thermal expansion coefficient to 20 folds [2]. Because sodium ions are so soluble in aqueous solution, calcium Oxide (CaO) is applied to the mixture in order to enhance its insolubility. Soda-lime glass is manufactured on a wide scale and used in bottles. Drinking glasses, windows. Its light transmission properties, as well as its low melting temperature, make it suitable for use as Glass window. Its smooth and non-reactive surface makes it excellent for food and drink containers. Recycled glass nowadays, often known as slaughtered, it is used to produce green glass, which aims to conserve electricity and reduce pollution [2].

1.5. Mechanical Properties of Glass:

The first efforts to research the glass began about 1880 focused on the glassy appearance. Instead of chemical or physical behavior, it was hard for scientists like Faraday and Kistler to describe the glass condition. Due to its fragile property, it was difficult to understand the glass state [3,4]. When researching the chemical and physical properties, the current theories rely on glass in the solid state. The complexity of glass structure is responsible for the difficulty of defining the glass features as a solid material. Heat treatment has a direct effect on glass materials when the glass is re-energized after cooling down, through melting silica, alumina and other oxides utilizing thermal therapy. Heat treatment and solidification are the main internal defects in the glass, which are the cause for failure of fractures and components under different loads [5]. The thermodynamic modifications of the glass composition during melting have an impact on the chemical properties of the manufactured material. Scientists understand the effects on the properties of manufactured glass of the heat treatment system used for glass processing. In the same formulation and under the same experimental conditions, for instance, the glass rupture modulus and expansion coefficient vary, emphasizing the analysis during formation of glass properties [6]. The degree to which the glass stress profile is articulated and projected during formation and based on the glass treatment method helps us to monitor the detailed properties of the finished product as a cumulative surface compression. During the transition of glass into solid state, expansion coefficient and depth of scratch and effect of the impact durability, these properties are formed. The framework restored during glass solidification, withstood the formation stresses and chemical compounds, which are meant to achieve balance, that solidifies the glass in a non-uniform way. Griffith studied the relation between the defects and the strength of the brittle material (e.g. glass), and the influence of surface energy on the propagation of fracture [7]. The strength of glass depends on the concentration and distribution of defects along the surface. Based on Griffith's findings, modern chemical strengthening methods attempt to cover and prevent these defects from expansion, thus increasing the strength of the glass.

1.5.1. Strength of Glass:

Theoretical strength of solids can be calculated in regard to [8]. with regard to elastic modules and surface energy:

$$\boldsymbol{\sigma_{th}} = \sqrt{\frac{\mathbf{E.\gamma f}}{\mathbf{ro}}} \tag{1.1}$$

Where:

E is young modulus of Elasticity (MPa)

γf is surface energy (J/m2)

ro is atomic material component equilibrium distance (m)Typical values

for soda-lime silicate glass: E = 70000 MPa

γf is 3.5 (J/m2)

ro is 0.2 (nm).

The theoretical value of strength results: σ_{th} is 35000 MPa [8].

This value is much higher than the trial value to soda-lime silicate glass, which is roughly 50 to 100 MPa.

The great disparity between laboratory and theoretical strength of glass values is due to defects on the surface of the glass. Stress amplifying faults amplify the application of the tensile stress at the tip of the failure to achieve the value of potential strength resulting in a breakage of glass. A simple idea because of Inglis should clarify how this can be done.

Consider an axis 2b and 2c elliptical fault in the effect of an applied stressσa. The stress on the tip is: in regard to Inglis 1913 (see Figure 1.2).

$$\boldsymbol{\sigma}_{\mathbf{yy}} = \boldsymbol{\sigma}_{\mathbf{a}}.\left(\mathbf{1} + \mathbf{2}\,.\frac{\mathbf{c}}{\mathbf{b}}\right) \tag{1.2}$$

The curvature radius at the end of the defect is

$$\boldsymbol{\rho} = \frac{\mathbf{b}^2}{\mathbf{c}} \tag{1.3}$$

And the results of the stress formula (in curvature radius):

$$\boldsymbol{\sigma}_{\mathbf{yy}} = \boldsymbol{\sigma}_{\mathbf{a}}\,.\,(\mathbf{1} + \mathbf{2}\,.\,\sqrt{\frac{\mathbf{c}}{\boldsymbol{\rho}}}\,) \tag{1.4}$$

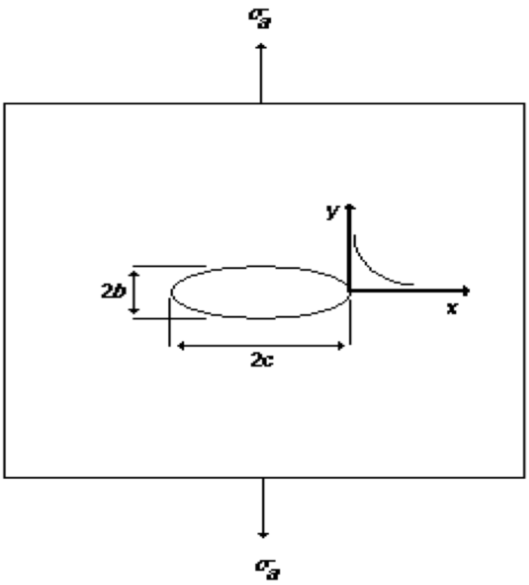

Figure 1.2. Elliptic failure in a glass sheet under the tensile stress applied σ_a [8].

If the elliptical defect (sharp flaw) is very long **c** >> $\boldsymbol{\rho}$:

$$\boldsymbol{\sigma}_{\mathbf{yy}} = \mathbf{2}.\,\boldsymbol{\sigma}_{\mathbf{a}}\,.\,\sqrt{\frac{\mathbf{c}}{\boldsymbol{\rho}}} \tag{1.5}$$

We may logically come to the conclusion that the diameter of curve ρ at the defect tip of the Si-O contract is of the similar magnitude in the suspected hypothesis with very sharp defects (ro). This makes it possible to define this condition of breakage from $\boldsymbol{\sigma_{yy}} = \boldsymbol{\sigma_{th}}$:

$$\sqrt{\frac{E.\gamma f}{r_o}} = 2.\,\boldsymbol{\sigma_b} \cdot \sqrt{\frac{c}{r_o}} \quad (1.6)$$

$$\boldsymbol{\sigma_b} = \frac{1}{2} \cdot \sqrt{\frac{E\,.\gamma f}{c}} = \sqrt{\frac{E\,.\gamma f}{4\,.c}} \quad (1.7)$$

Where σ_b is the stress of breakage. Considering the size of the flaw of 10 μm, the following results: $\sigma_b = 78\ MPa$

The final strength of a 50 μm severe fault will be: $\sigma_b = 35\ MPa$

All results are very close to laboratory standards.

A thermodynamical justification [8]. can be used to draw a different strength requirement, since the defect needs to reach a certain length, at which free propagation will occur. A total energy (**U**) of the device with an Exercise bike Length Defect **2c** is included in this criteria. In the elastic environment, the elastic stress energy per unit volume (J / m3) is only the area under the tension - strain curve determined by Hooke rule (see figure 1.3)

$$\boldsymbol{\sigma} = \mathbf{E}\,.\,\boldsymbol{\varepsilon} \quad (1.8)$$

Where : $\boldsymbol{\sigma}$ =stress , $\boldsymbol{\varepsilon}$ = strain , $\boldsymbol{E}$ = young modulus.

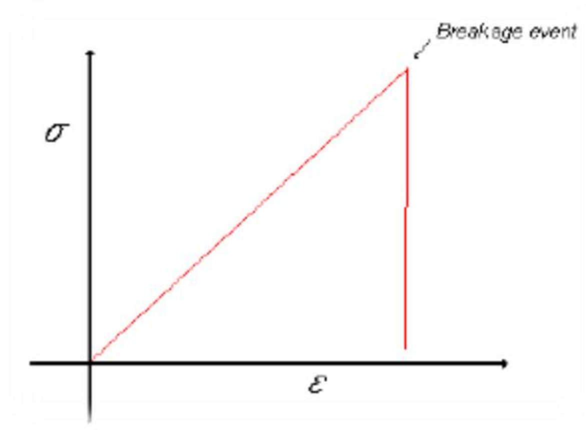

Figure 1.3. *Stress – strain curve.*

This is 1/2 $\sigma.\varepsilon$. taking into account an infinitely thin elliptical fault (b → 0), this energy is shared across a fault surface, and the flaw introducing a appropriate geometric Factor Y can be explained as elastic strain energy by unit length **c**. Finally, the elastic strain energy can be defined per unit length from the crack front (J / m).

$$\mathbf{U_{el} = -\sigma.\varepsilon.Y^2 . c^2 = -\frac{\sigma^2}{E} . Y^2 . c^2} \tag{1.9}$$

In case of a length **2c** defect that increases because of this stress, this elastic stress energy per unit of length is greatly reduced to create two new defect surfaces, which require an increase in surface energy per unit of crack length (J / m):

$$\mathbf{U_s = 4 . \gamma_f . c} \tag{1.10}$$

Taking into account the stress applied ($\boldsymbol{\sigma = \sigma_a}$), a unit fault length of the total elastic energy is generated.

$$\mathbf{U = -\frac{Y^2 . c^2 . \sigma_a^2}{E} + 4 . c . \gamma_f} \tag{1.11}$$

This means that when the applied tensile stress is sufficient to create new surfaces the crack spontaneously spreads, otherwise the crack remains in its initial length.The first term (1.10) refers to the reduction in stress energy used it to generate new surfaces, which represent a surface energy gain (second term (1.11). For **(c)** values the second term prevails as well as **U** is positive, however as c second term is larger, the first term prevails and **U** is constantly reduced as (**c**) increases. A correspondence at the (**U**) maximum amount is the critical value for flaw length ($\boldsymbol{c}^*$) which it grows spontaneously and it can easily be seen to distinguish (**U**) in relation to (**c)** and set the derivative to zero:

$$\boldsymbol{\sigma_b} = \frac{1}{Y} \cdot \sqrt{\frac{2\,.\,E\,.\,\gamma_f}{c^*}} \tag{1.12}$$

For soda-lima glass, it is possible (as an example) using this latest criterion as a geometric factor of Y= $(\pi)^{1/2}$ and a flaw of 10 µm: $\sigma_b = 124\ MPa$

The final strength of a flaw of 50 µm is the following:

$\sigma_b = 56\ MPa$. This last value is very similar to the experimentally identified values. This analysis shows that the strength of the glass is strongly linked to the surface state by the presence of micro-fissures. These defects generally extend into the glass, from a few to ten microns. Few could be even harder, with the result that mechanic strength is greatly reduced (see Figure 1.4).

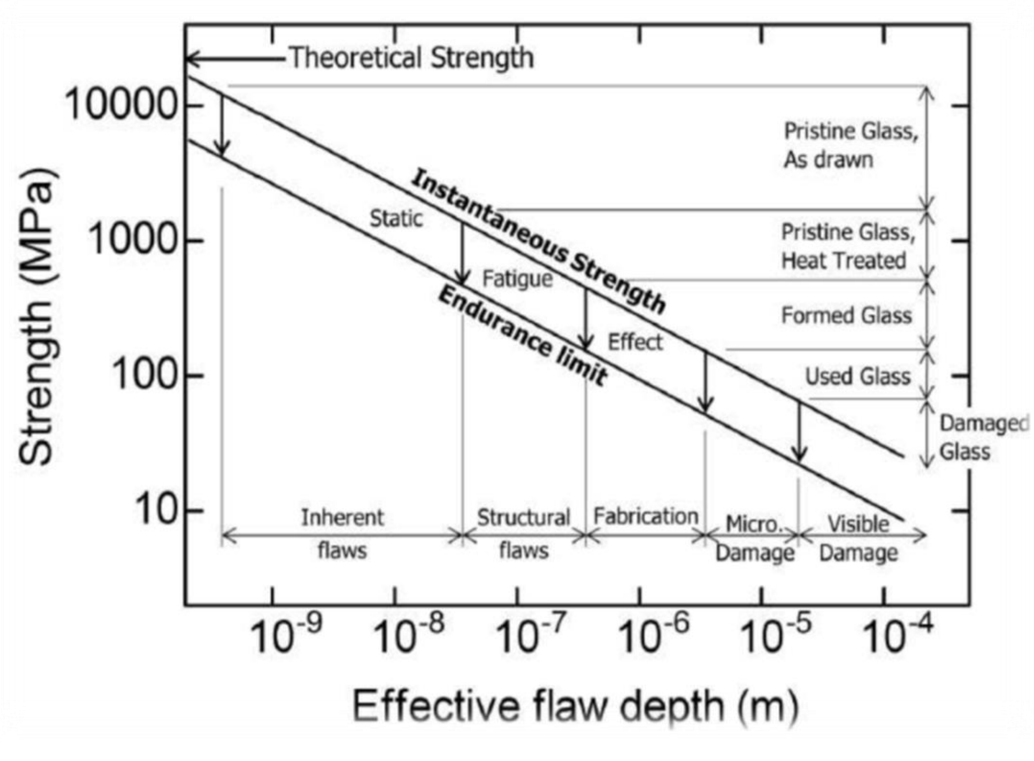

Figure 1.4. *Flaw depth and Glass Strength [9].*

A number of sources may have flaws. The first source is the production process itself for glass items: contact with the handling equipment and particles and atmospheric pollution can cause glass surface defects. Faults in glass objects can be created even more subsequent to the processing as cutting, chamferıng, lamination, packaging. The result is a glass item with a large variety of defect lengths (surface population) that match a variety of rupture forces (figure 1.5) [8]. According to this argument, a statistical concept strongly connected to statistics of surface defects can easily be found in the glass strength.

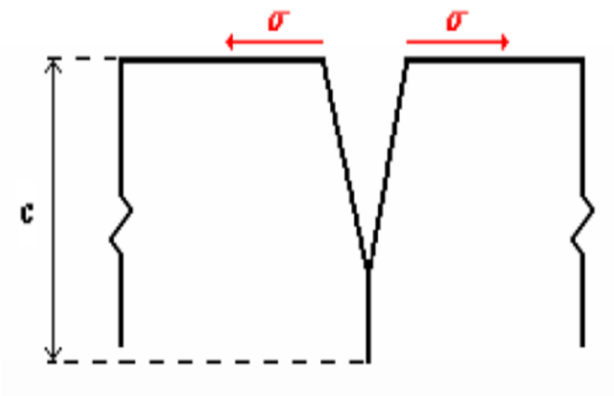

Figure 1.5. *Schematic depiction of a surface defect under tensile stress [8].*

It can be seen from calculations (1.7) and (1.12) that the condition of breakage can be simply expressed as:

$$\boldsymbol{\sigma_b = Y \,.\, \sqrt{c} = \sqrt{2\,.\,E\,.\,\gamma_f}} \tag{1.13}$$

When E is a material and Yf is depend on the material and the environment.

In terms of concept of fracture mechanics that introduce the stress intensity factor **(SIF)** $\mathbf{K_I}$, the equation can be arranged. It is a factor related to the stress applied and the defects (geometric factor **Y** and great depth **c**), and is the first equation factor (1.13). Afforded a defined surface flaw characteristic than the SIF is proportional with the stress used, as follows:

$$\boldsymbol{K_I = \sigma_a \,.\, Y \,.\, \sqrt{c}} \tag{1.14}$$

This equation will be turned into the equation of the disruptive condition that defines a critical stress factor $\mathbf{K_{IC}}$

$$\boldsymbol{K_{IC} = \sqrt{2\,.\,E\,\gamma_f}} \tag{1.15}$$

Fracture condition occurs as $\mathbf{K_I} \rightarrow \mathbf{K_{IC}}$. This condition is achieved by increasing σa or increasing **c**. Units for $\mathbf{K_I}$ are accordingly [MPa · $m^{1/2}$]. From these definitions it is clear that $\mathbf{K_{IC}}$ is a material property slightly influenced by the environment because of (γ_f).

In table (1.1) some typical values are reported for the critical stress intensity factor (**K_{IC}**) and Young modulus (**E**) for glasses of different chemical compositions.

Table 1.1. *Young module and stress factor for various glasses [8].*

Glass	E (MPa)	K_{IC} (MPa.m$^{1/2}$)
Fused Silica	73000	0.74 – 0.81
Soda-lime silica	7000 - 74000	0.72 – 0.82
Aluminosilicate	83000 – 91000	0.85 – 0.96
Borosilicate	64000 – 89000	0.75 - 0.85
Lead-silicate	58000 - 65000	0.62 - 0.73

In fracture mechanics, the component of stress strength, depending on the crack extension method, is three essential values (see Figure 1.6):

1. Tensile (**K_I**) opening where the failure plane and the stretch is perpendicular to the central force,
2. Sliding with shear failure (**K_{II}**), if the stress applied is behaving in the propagations direction on sliding fracture planes,
3. tearing in shear (**K_{III}**) where direction of propagation is perpendicular to that of thestress but failure plane is parallel to shear stress direction.

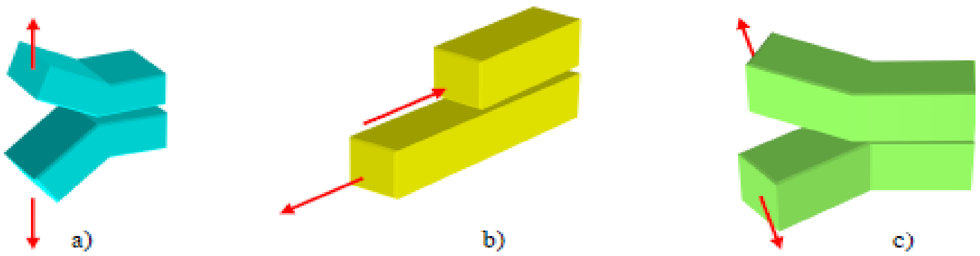

Figure 1.6. *Crack extension modes: a) K_I, b) and c) K_{II}, K_{III} . [10].*

Elastic stress energy is described in the research by the letter G and equal to K2/E for each unit crack duration Uel. G is thus the change in the body's strain intensity for the crack length move. The Griffith criteria (1.12) is used for our case (Y= π):

$$\mathbf{G_{IC}} = \frac{\mathbf{K_{IC}^2}}{\mathbf{E}} = \frac{\mathbf{\sigma_b^2 \,.\, Y^2 \,.\, C^*}}{\mathbf{E}} \tag{1.16}$$

And:

$$\boldsymbol{\sigma_b} = \frac{\mathbf{1}}{\boldsymbol{Y}} \cdot \sqrt{\frac{\boldsymbol{G_{1c} \,.\, E}}{\boldsymbol{C^*}}} \tag{1.17}$$

This leads to the $\mathbf{G_{IC}}$ definition:

$$G_{IC} = 2\,.\gamma_f \tag{1.18}$$

The toughnes is ability to absorption of strain energy befor failuring. Toughtness is area under curve, given by the curve of stress / strain. This is of no significance in the macro scale for a brittel solid. The energy needed to extend a defect from its original to critical length should be considered. G_{IC} may therefore be called a measure of the toughness of fracture. Some authors call the factor of critical stress intensity

$$K_{IC} = (E\,.\,G_{IC})^{1/2}$$ "fracture toughness of material".

1.6. Types of Glass Strengthenıng:

1.6.1. Thermal temperıng of glass:

Thermal toughning involves cooling the glass quickly from temperature above Tg, i.e. a quenching the glass with air, liquid or fluid air. The surface cools faster than inside, and at a certain point. The argument the surface is going to be an elastic base when the thermo-elastic stress relaxation can still be experienced in the interior. Thus, the surface occurs in the interior contraction. It's in a compression state. The Balance of tensile stress is being developed inside the glass and is normally half the compressive value. Stress. The stress profile achieved is parabolic as sgowing Figure (1.7) [11,12,13]

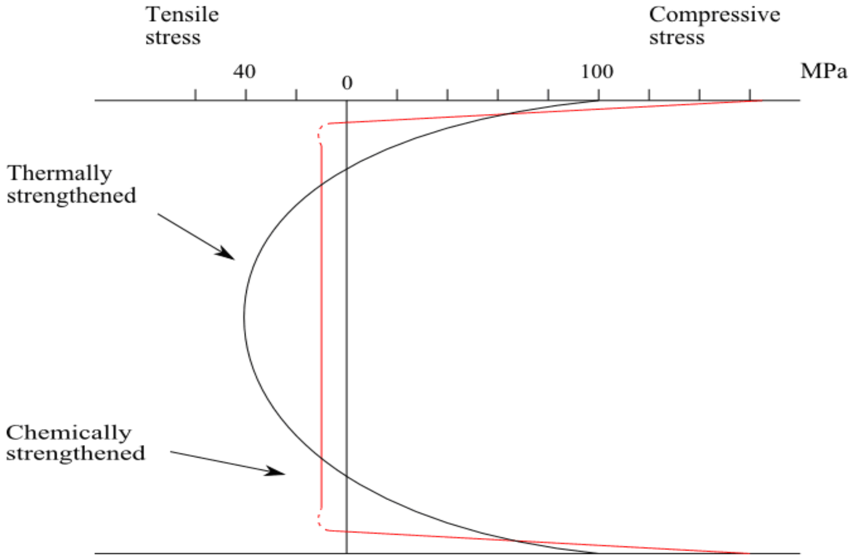

Figure 1.7. *Comparison between the residual stress distributionsin thermally and chemically strengthened glasses[11].*

1.6.2. Propertıes of thermal temperd glass:

Tempered glass is heavier than standard glass mechanically and thermally[11]. Greater outer layer contraction during processing results in compresive stress on the glass sheet, offset by tensile tension on the glass core. The compressive surface tension will surpass 100 megapascals if it is to be called tempered glass. Due to the elevated surface tension the glass splits into tiny round pieces, rather than large jagged fragments. The glass is still broken. This function renders the tempered glass suitable for applications that are high-pressure and explosion-proof[12]. This compressive force improves the strength of the tempered glass. Annealed glass with virtually no interior friction produces tiny surface cracks and any stress on the surface induced by the lack of surface compressions will contribute to a crack distribution. When a crack begins to grow, the stress becomes intensified more at the edge of the crack, which allows the crack to expand at the sound level. Annealed glass is often brittle and is broken into small, sharp pieces[13]. The compressive force on a tempered glass on the other side causes the defect which inhibits its proliferation or extension. Cutting or grinding before tempering must be carried out. Upon tempering, the glass is shattered by the cutting, scratching and harsh impact.

1.6.3. Uses of thermal temperd glass:

Tempered glass is used according to its mechanical properties, thermal resistance, strength and safety, all three specifications involved in commercial vehicles for example. Since it is used mainly outside, it is exposed to constant heating, cooling and dramatic weather changes, in addittion it should withstand damages from small stones and vehicle collisions. Since big, sharp fragments of glass present an unnecessary and inacceptable passenger danger, tempered glass is used to render the pieces blunt and relatively harmless when shattered. Usually, the windscreen is composed of laminated glass, and when shattered it smashed into small blunt fragments. Other applications like:

(Athletic facilities, Balcony doors , Swimming pools, Bathroom and Shower doors

Exhibition areas and displays).

1.6.4. Disadvantages of thermal tempered glass:

tempered glass must be cut to size or shaped in order to fit and not reworking as soon as it is tempered. Before the tempering process starts, edges or boiling hole are polished or chamfered in glass. According to the balance stresses in the glass, every component of the glass can gradually become smashed because of fracture. The glass is more vulnerable to breakage at the glass edges, where the stress force is more, eventhough it can be damaged anywhere if the force is focused on a point[14]. In some cases the use of tempered glass may present a safety danger due to the potential to break completely on a rough impact instead of holding fragments in the window frame. After it has been shaped using this method the surface of tempered glass shows waves at surfaces. Which means destroying its optical propreties[15].

1.7. Lamination Method:

Laminated glass is a kind of protective glass, which holds intact when smashed. In case of a break, it is retained between the two or three layers of glass by an interlayer, usually polyvinyl butyral (PVB) or ethylene-vinyl acetate (EVA). The polymers connects the layers of glass even though they are separated and avoids the glass being split into small parts. It creates a distinctive cracking pattern for the "spider web" where the effect is not enough to pierce the glass entirely. The thermoset EVA offers a complete bonding (cross-linking), whether it is polycarbonate (PC), or other component kinds, with the glass. Laminated glass is typically used where a human safty is likely, and often for art. Usually, aircraft and vehicle covers utilize laminated glass. Laminated glass is also used for exterior storage frames, curtain walls and windows for areas requiring hurricane-resistant design. Laminated glass is often used to enhance the acoustic insulution of a frame, and increases the acoustic insulution greatly relative to unlaminated glass panes with similar thickness. A specific "acoustic PVB" compound for the interlayer is used for this purpose. No extra acoustics are needed for the EVA content, as the EVA offers sound insulation. An unusual advantage of the window lamination is that the most ultraviolet radiation may be effectively blocked with PVB and EVA interlayer. Up to 99.9% of all UV rays will be filtered by an EVA thermoset

1.8. Chemical Strengthening of Glass:

Chemical Strengthening of Glass is an increasing strength form of glass arising from the chemical process in post-production. When it's split, it shatters in long splinters like spinning plates. This is why a safety glass is not included and should be laminated if a safety glass is available. But, However chemically strengthened glass usually is about 6-8 times of float glass strength. Gorilla glass is the most popular brand of this type of product. A surface finishing technique chemically strengthens. Glass is immersed in a potassium salt bath at 300 °C [1]. It immersed in a bath solution mixing sodium ions on the glass surface with potassium ions. This causes sodium ions in the glass surface to be replaced by potassium ions from the bath solution. These ions of potassium are larger than sodium ions and wedge into the gaps of the smaller ions of sodium when migrating to the solution of potasium nitrate. This substitution of ions causes the glass surface to be compressed and the core to compensate for tension. Chemically tempered glass surface compression can reach a maximum of 690 MPa[1]. The strengthening function relies on the significantly greater intensity of compressive strength of the glass than its tensile strength. For all glass surfaces being under compression now, it takes an incredible amount of bending to be fractured. To reach tensile strength, further bending is required. The other surface is under rising compression. But as the compressive strength of the glass is much higher, there is no compressive failure. A two-stage, more sophisticated method for manufacturing chemical strengthened glass is also usable, in which the glass is first dipped into a 450 °C sodium nitrate bath that makes the surface enriched by sodium ions. For the immersion of potasium nitrate, more sodium ions are added on the surface to be substituted by potassium ions later. This increases the potential to compact the surface of the finished piece by using a sodium nitrate wash. The chemical tempering results in a strength equal to concrete. But there are no extreme temperature fluctuations in the phase and therefore chemically tempered glass has little or no bow or curl, optical distortion or strain pattern. It varies from thermal tempered glass, which greatly bows at surface. Chemically tempered glass can also be cut after tempering, unlike thermal tempered glass, but loses its strength within the region around 20 mm from the cut, similarly, if the surface of chemically tempered glass is heavily damaged. Although the method of making thermal tempered glass is easily available, chemically temperd glass has a significantly more expensive business path. Such costs prohibit the product from being used in various applications. For the aircraft canopy of fighter aircraft, chemically temperd glass was used.

1.8.1. Types of chemical strengthening:

- Etching process: In this technique surface flaws and damaged areas are removed using chemical etching, chemicals used to form the etching solution are a dilution of aqueous solution of hydrogen fluoride, hydrochloride or Sulfuric acid. The solution leads to a reaction between hydrogen fluoride and glass, leading to removal of flaws from the glass surface or increasing the cracks tips radii [3]. Bulk glasses can be processed by this method and produce a mechanical strength over 1000 MPa.

- Thin coating process: Thin coating is focused on a chemical layer which covers a glass surface, which is able to stick to the surface and protect the surface against scratches or indentation. The titania and tin oxide are some of the coating products used for this method[16].
- Cladding process: coating surface with coat having a lower coefecient of expansionm making compression at the surface and improve the strengthening of glass.
- Ion exchange process: This study focuses, as already mentioned, on this technique. Briefly by exchanging small ions from the glass with larger ion, having same equivelent numbers, Like potassiom or lithiuom.

1.8.2. Ion exchange technique:

Using this description, the ion exchange processes carried out under the transition temperature of the glass. Ione exchange is not a thermodynamic equilibrium cycle according to its design. The ion flux in and out of glass patterns is the mechanism guided by the electrochemical potential gradients of the ions. Also it can be defined as follwing: “Ion-exchanged glass belongs to the class of “forbidden glass” i.e., a glass with a structure that is not attainable by cooling from a melt using any possible cooling path”[17]. Let us presume the following definition: the silicate glass Ion Exchange (IX) is a non-equilibrium thermodynamic mechanism between the glass and the ion source powered by gradients in the comparatively mobile network modificators (electrochemical capacity). This includes a glass matrix with fairly moving ions (typically monovalent alkali) and an ion source with freshly produced mobile ions to enable ion exchanges. The interaction of this glass matrix with the ion source results in a gradient difference in the electrochemical potential of the concerned ions. The Si-O glass network may be considered stable throughout the ion exchange cycle. With respect to ion (A) from the ion source (IS) and ion (B) from the glass matrix (GM), the IX cycle may be written as:

$$A_{IS} + B_{GM} \leftrightharpoons A_{GM} + B_{IS}$$

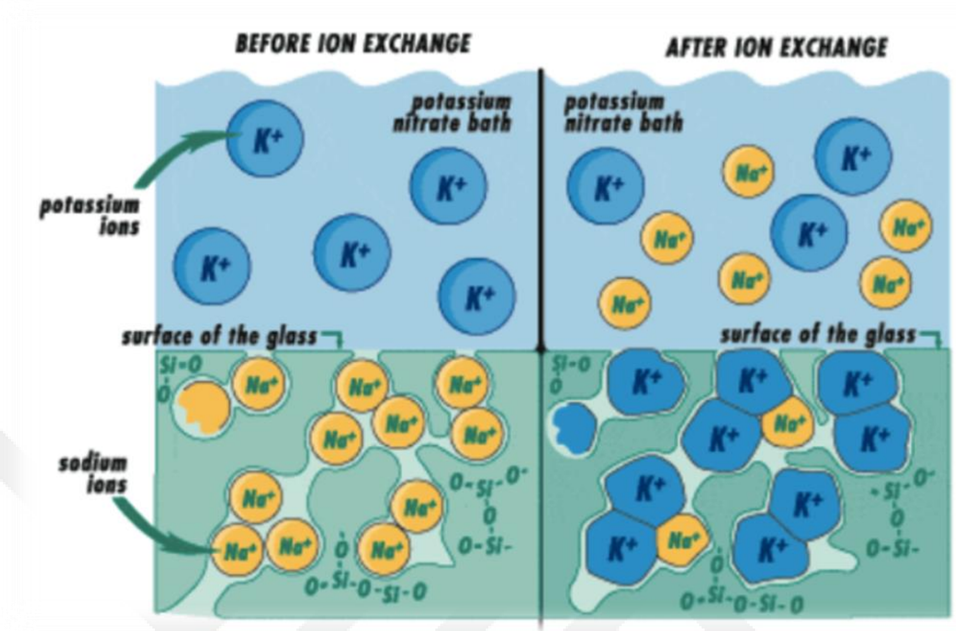

Figure 1.8. *Schematic of Ion exchange process [1].*

In the industrial sector, ion exchange is done via a large container of molten salt made of stainless steel above the melting point of used salt in the reaction and below the glass temperature of transition. The potassium ions began to interact on the glass surface, as shown in Figure (1.8), as rapidly as salt (eg potassium nitrate) reached the surface of the glass at high temperatures of the oven. One of the most popular ion exchange salts of the industry is Potassium nitrate. Glass items are left for several hours (e.g. 2 to 16 hours) inside the stainless steel vessel. The glass comprising sodium or lithium silicates is the most widely produced form of glass[16,18].

1.8.3. First successful ion exchange technique:

In 1962, Kistler defined "Chemical Strengthening" as a "successful strategy," particularly "Ion Exchange Glass Strengthening"[3]. The scientific paper identified a toughning of glass enhancement method and the main topic discussed was the propagation of interdiffusion effects on glass. It was reported, depending on the processing time of the glass article, that the strength is doubled and sometimes tripled.

1.8.4. Advantages and disadvantages of ion exchange techniques:

In several papers [4,19], from different perspectives, the drawbacks chemically tempered glass by ion exchange were addressed. The drawbacks recorded in the ion exchange methodes are the long glass processing period, that ranges from 1 to 12 hours depending on the strength required and the types of alkalis implicated in the exchange process. Ion exchange process profit has been recorded by researchers. These include the flexibility of the form of the glass to be processed, no thickness restriction of the glass and no thermal expansion coefficient limitation. It led to the involvement of many companies in the industry. In today's communications devices, they converted technology into air craft canoby windshields, photocopiers in glass, strong power to weight glass containers and fine screens[20].

1.8.5. Factors affecting ion exchange efficiency:

1.8.5.1. Salt bath impurities:

The quality of the ion exchange method directly affects the toughning of the glass. Some researchers report that the impurities of salt bath impact on the network of surface glass results in undesirable results of the toughning procedure[21]. One of the solutions recorded for salt bath impurities is the use, in an ultra clean, stainless steel tank, of a very pure and high-concentration salt to minimize impurities of salt bath and double the performance of ion exchanges, however these pure salts are not economically feasible, which have driven scientists to consider alternative low cost and same performance. Some researchers have reported that salt impurities improve efficiency of the ion exchange, but this claim does not depend on well-defined experimental results or any empirical quantitative knowledge on the chemical and mechanical methods used to obtain the result [22]. It is previously known as (process poison) that certain alkaline and especially alkaline earths obstruct the effectiveness and reduce ion exchange. Zhang et al., deeply discussed the effects of impurities in molten of potassium nitrate solution in the process of exchanging ions and considered some of them as impurities as they block or reduce the efficiency of ion exchange (Figure 8), especially, the presence of calcium ions. The exchange process is blocked by the solution. Zhang et al. performed a study of the influence of the potassium and sodium ion exchange from various alkaline earths. They noticed that the alkaline earth molar percentages at which the blockage effect begins and their study concluded that the effective blockage rate in the molten salt (0.01 mol percent) is for calcium ions (Ca). Strontium (Sr) and Barium ion (Ba) were other impurities that impede the chemical strengthening of glass cycle but the percentage of the molars necessary must be greater. This suggests more barium and strontium should be condensed in the salt bath to have an impact of blocking significant to remember here (0.1 mol percent of Sr and Ba ions, Figure (1.9)) [23].

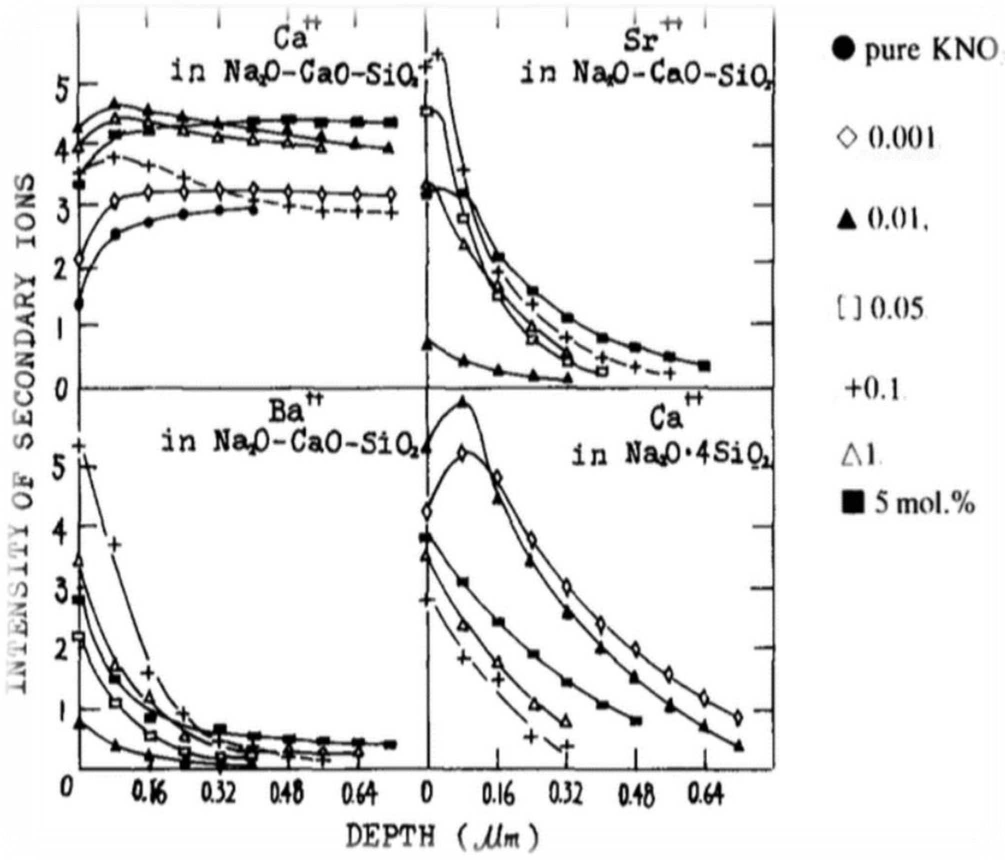

Figure 1.9. *Intensity of secondary ions -depth profiles [23].*

Figures 8 above shows different formulations of $Na_2O.4SiO$ glass and the profiles of various molar percentages of impurities at various depths and the resulting effect on the sensitivity of potassium ions. It concludes, that even with small molar amounts, including Ca, Ba and Sr ions as seen in Figure (1.10), the ions for impurities with a bigger radius that significantly affect the diffusion.

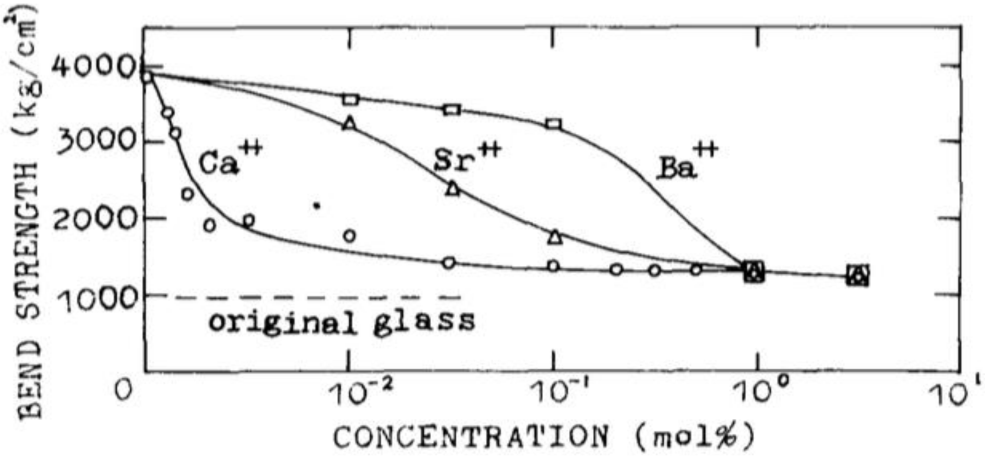

Figure 1.10. *Bend strength-concentration(mol%) [23].*

The calcium impurities affect the strength of the glass with greatly reduced strength compared with strontium and barium ions, as these ions hinder the distribution of potassium ions and the divalent diffusion of the glass surfaces.

1.8.5.2. Chemical composıton of glass:

The composition of the tempered glass has also direct effect on the mechanism of ion exchange, which several researchers note. Several specific glass formulations of a particular percentage were tested to determine the effect on glass strengthening process.

1.8.6. Multi-stages ion exchange:

Considering that each phase of ion exchange has its own specific effects depending on the properties of the ions exchanged, scientists have attempted to study ions over many phases, which match the ions ideally adapted to the treatment of glass [22,24].

The glass has treated with a several alkaline ion exchange to improve one solution. The process is used in lithium-soda and glass of soda-lime silicate. The structure of the soda-lime silicate includes sodium and potassium ions. The structure of glass has a significant part in the performance of the ion exchange procedure and is often considered a crucial factor [22]. The alkalis that are exchanged should be high in concentration compared to ions concentration in glass structure. Except when strongly diffusive ions are used, the ions may be diffused across the salt bath through the glass surface at low concentrations. In specific, ions with a smaller atomic diameter may be substituted better with lower energy than ions with a larger atomic diameter, allowing greater penetration through the surface of the glass[22]. Two steps are required for the multi-stage ion exchange. The first is filled with lithium nitrate ($LiNO_3$) and potassium nitrate (KNO_3), and the soda lime glass sample is plunged into the molten bath at below-glass temperature. It refers to the interaction between lithium and potassium ions on the glass surface with sodium ions. In the second phase you clean the glass and wait for the glass to cool down gradually in the lithium salt water. In this case, lithium is substituted for sodium ions, and the glass is then sunk into a potassium nitrate bath where potassium ions are substituted for sodium ions on a glass surface. We note that only the sodium ions on the glass surface are exchanged when we exchange the lithium ions with sodium. Although the lithium ion radii (0.076 nm) is comparatively shorter than the radii of sodium ions (0,102 nm), Figure (1.11) indicates fairly large density and size of the lithium ions. This means that the sodium ions on the surface are substituted and replaced with lithium ions for a greater depth compared to potassium, which is exchanged with only sodium ion, this will increase glass strength by substituting a fairly significant volume of sodium ions from the surface.

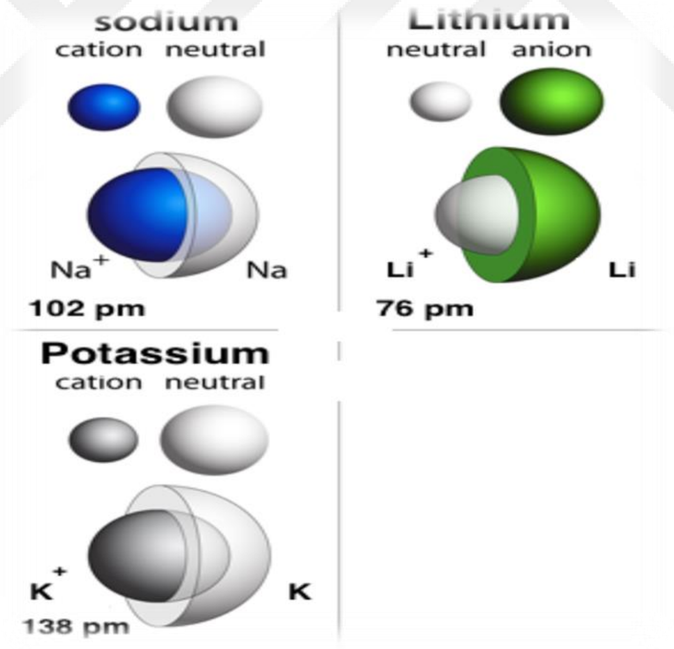

Figure 1.11. *Na, Li and K radii illustration [22].*

While lithium is used much in the multi stages in ion exchange (i.e. lithium-soda glass) by substituting smaller lithium ions for larger sodium and potassium ions, this process is also applicable to many forms of glass containing alkalis in their composition by replacing one form of ion at a time and then the other ion (potassium) is exchanged. The temperature is the key element in the experimental portion of this method. Iones with a greater ionic radius substitute ions with a comparatively smaller radius, while smaller ions replace a fairly big ion at a marginally higher phase temperature than the strain temperature [22]. Saunder et al. identified a new concept in their study that before a multi-stage exchange of ions is carried out, a modified glass formulation is needed, and this modification greatly increases the impact of an ion exchange and hence the strength of the glass. The following table (1.2) shows one of the recommended compositions of the tempered glass by Saunders et al.

Table 1.2. *Customized glass composition of based glass [22].*

Oxide component	Percent by weight
SiO2	44.38
Al2O3	26.61
Li2O	5.04
Na2O	11.0
P2O5	9.69
ZnO	3.0

The author reported that this mixture consisted of four forms of molten salt utilizing each other of 4 x 4 inches by (1mm) thick glass slide, which later are submerged in an alkaline metal molten salt bath. The product of the sample was defined in the following table 1.3

Table 1.3. *Four ion exchange treatment of customized glass samples [22].*

Molten salt	Temprature of ion exchange (ºF)	Treating time	Total compression layer depth	Surface compressioı Lbs, PerSq. In.
$NaNO_3$	850	30 (min)	200 μm	60.000
KNO_3	925	do	18 μm	96.000
KNO_3+$NaNO_3$	875	do	215 μm	92.000
KNO_3+$NaNO_3$	875	do	185 μm	98.000

As can be shown in Table (1.3) the individual glass composition is processed at a temperature of (496 °C) and 30 minutes with potassium nitrate molten salt water. The complete depth of the compression layer and a surface compressive value of 660 MPa were obtained during this treatment. This finding demonstrates how necessary it is to preserve a low depth of the sheet. As the broad importance of layer depth is recognized in the prior art, not inherently contributes to a higher compression, but much of the time to stress relaxation. Potassium nitrate correct stoichiometry and a fair treatment period together led to a strong surface compression rate were the explanations for effective glass strengthened in this experiment.

1.9. Stress Relaxation:

The goal of glass strengthening is to obtain the highest strength of glass. However, when the authors were handling glass at higher and longer temperatures, they sought to improve the procedure by determining the ideal thermal temperature and period to obtain the best potential strength of the material. They found during the experiments that the strength values of the glass hit a high point and then the strength drop rapidly. It is called as stress relaxation. Stress relaxation is occuring at variable enviroments and in different forms. It makes it difficult to explain the reason behind this phenomenon because relaxation takes place close to the stress peak values and immediately after the completion of the ion exchange process. It was first created by the diffusion of larger ions through the glass sheet. In order to create fresh ions sites, the glass network restructures. This process of restructuring leads to elastic deformations caused by the removal and advent of smaller ions on the surface of the glass. More studies also noted that this relaxation is induced by constant plastic deformation triggered by the modification and change of unbridged oxygen and by transferring or shifting bridge-. This relaxation mechanism is known as CIRON (Cation induced relaxation of the network). CIRON starts with the slower invasion of large ions and the restoration of the network, Figure (1.12) [5]. Stress profile is the descriptive method for glass strength, and the compression of the surface decreases by increasing the exchange time, but meanwhile, the penetration depth for diffused ions increases [5]. As the differences in thermal expansion coefficient of the glass composition also generate compressive layers, the thermal history of glass has an influence on the possibility of stress relaxation. The only approach for researchers to prevent strain relaxation was by the method of ion exchange clearly far away from the transition glass temperature and at the same time by an exchange in a suitable period to obtain the maximum effects from the strengthening process.

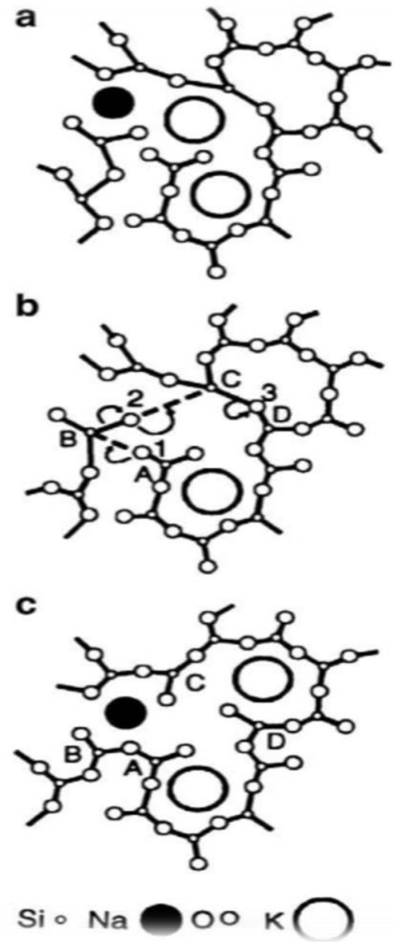

Figure 1.12. *Cation induced relaxation of network (CIRON) [23].*

As shown in Figure 1.12, the bonds are able to extend or curve in the microscopic structure in the field of ion exchange in order to accommodate the freshly introduced ions inside the glass structure, which are meant to create a permanently stable ion position. Physically the volume will be increased with this change, but the glass structure adapts to adjust the volume in order to accommodate the new ions, thus keeping the form intact, and we can imagine the complicated estimation of the position of the new ions in the configuration of the glass structure realizing that every part of the glass structure has an elastic limit which is different from others.

1.10. Diffusion:

In the glass surface, the process of the diffussion of ions and their kinetics explain how deep ions enter the material. The main factor in the process of ion exchange is diffusion. This measure the ability of the ions to move and interact with the various components of the glass system. Interdiffusion is the basic cause of diffusion and is applied generally to glass chemical strengthening and more precisely to the ion exchange of chemical strengthening. Interdiffusion happens here between entrant ions and the ions on the surface of the glass. In the same time, the diffussion of ions inside glass structure. The ions interact with sodium in solution when the glass specimen is submerged in a melt and/or a solution of monovalent ions. The diffusion control rate on the glass surface should be investigated to clarify the ion exchange [25]. Quantitative equations, such as Fick's equation, are important tools to provide a complete explanation of the diffusion mechanisim. If various atoms are involved in diffusion, an equivalent diffusion coefficient may be reached as follows:

$$\mathbf{D} = \frac{\boldsymbol{D_1\,D_2}}{\boldsymbol{N_1 D_1 + N_2 D_2}} \tag{2.1}$$

where, D: is coefficient of interdiffusion, N: is local ionic fraction.

during ion exchange Na- K ion interdiffusion coefficients are observed nearly constant and irrespective of the local level and exchange period for soda lime glasses [5].

Equation (2.1) can therefore be translated as Na-K:

$$\mathbf{D} = \frac{\boldsymbol{D_{Na} D_k}}{\boldsymbol{D_{Na} N_{Na} + D_K N_K}} \tag{2.2}$$

Where N_1: is the alkaline ion fractional concentration, and D_1, is the mixed alkali glass composition diffusion coefficient. Equations (2.1) and (2.2) are known as Fick's diffusion law and Nernst-Plank equation respectively. In the previous literature, it is established that interdiffusion accelerates with inner tension stress and decreases by surface compression. The association between ion diffusion on the surface of glass and stress relaxation is possible. Many researchers conducted experiments with different compositions of glass and provided diffusion effects as figures to demonstrate various diffusion activities with different ions exchanged, as in figure (1.13) [26]. The figure describes the relation of the fractional exchange (γ) and the coeffecient of diffusion, when the difference is clear between sodium diffusion (Na) and potassium ions (K) and interdiffusion between the ions exchanged. Figure (1.13): Variance of the interdiffusion coefficient in relation to the ion-exchanged zone mixed-alkali ratio[26]. This figure comes with the calculation of data from custom glass samples, some of which are mostly soda lime silicate glass and others are samples of sodium borosilicate glass.

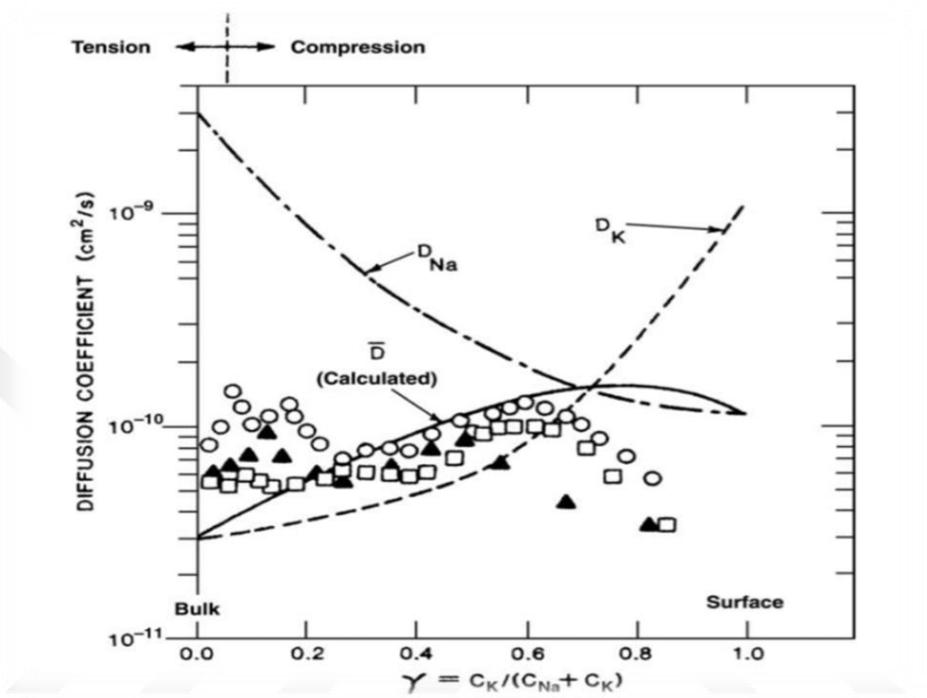

Figure 1.13. *Variation of the interdiffusion coefficient as a function of the mixed-alkali ratio covering the ion exchanged region [2]*

1.11. Armor Plate Glass:

Glass/polymer-laminated bulletproof materials are widely used for windows or windshields of military vehicles, battleships and warplanes because they are lightweight, transparent in visible light, and have good performance. However, the ballistic impact resistance of glass / polymer materials is often lower than that of metal (steel, aluminium alloy, etc.) and ceramic (aluminium oxide, silicon carbide, boron carbide, etc.) due to its low density [27-32]. It is therefore necessary to develop thin, lightweight and transparent bulletproof materials with higher ballistic resistance and improved operational capability in order to protect human life and valuable equipment. Traditionally, clear ballet-proof windows are made by laminating elastomer-like polycarbonate (PC) on monolithic glass using polyurethane (PU) and by linking the various thicknesses of the glass using polyvinyl butyral (PVB) [33], [34]. While the glass is broken after the impact energy of the projectile, the PC decreases the speed of movement of the projectile, avoiding plastic bulge penetration. Walley et al. [35] studied crack and spall forming within the PC during impact. They reported ways to prevent penetration and spread of cracks by varying glass/PC laminate construction. The production of better clear bulletproof windows is being widely studied through consideration of laminate construction, interlayer bonding technology, polymer composites and sandwich structure. Sabet and Bobaru [36,37] examined the ballistic impact resistance of glass / polymer laminate of differing densities and addressed the importance of parameters such as impact strength, density and ballistic limits. Grujicic and Gu [38,39] examined the effect of cracking on the glass during the attack. Through testing the ballistic resistance of transparent armor glass using a finite element technique, it was established that defects in the inside of the glass and fractures that existed during the cutting and grinding process contributed to damage to the bulletproof coating and decreased ballistic resistance. Rouxel and Sellappan [40,41] carried out valuable work on the enhancement of surface cracking resistance in various glass formulations using normal single-edged bending fracturing resistance measurements. These results can be connected to many important factors that affect ballistic resistance such as relative length, laminate length of glass and polymer (i.e., PC, PVB and PU) and ballistic limit speed (V50), depending on the stacking series, to boost the efficiency of protection [42-44]. In order to assess the impact resistance according to the different properties of glass, different criteria must be taken into account. However, due to the additional machining, polishing and strengthening that occurs before lamination of materials the determination of impact resistance is complicated. The mechanical properties, including the hardness of Vickers, the fractures toughness and the strength of the fold, are nevertheless essential considerations in terms of ballistic performance. Glass strengthening methods such as hydrofluoric acid etching [45], heat treatment [46], crystallization [47], and ion exchange are essential [48][49]. The consistency of the glass surface usually influences the optical and mechanical properties. Several types of glass materials have been used for transparent bulletproof windows, such as SLS, borosilicate glass, sapphire windows, etc. [38,40]. Sapphire windows typically have excellent mechanical properties, but because of the absence of alkaline ions, they are hard to produce, and chemical strengthening costly [49]. Also, ion exchange with borosilicate glass was reported to be difficult for its low alkaline content (4 mol% of Na_2O) and is a long process. SLS glass, however, can be easily replaced by ion, as the alkaline content is higher (14 mol percent Na_2O), compared to other materials, it is also less expensive. Thus, the ion exchange method may be used to manufacture transparent bulletproof materials if the maximum penetration depth is obtained. Throughout this study, the ballistic maximum rate, impact absorption capacity and basic energy absorption of bulletproof materials laminated with strengthened soda lime silicate SLS glass and polymer film were measured in terms of width, region density and stacking series [47-50].

2. EXPERIMENTAL AND CHARECTERAZITION

2.1. Experimental Procedure:

2.1.1. Preparation of glass:

Two types of soda lime silicate used in this study. The Glass was tested by XRF to determine the composition of glass, the XRF results of type 1 (thick glass 4mm) and type 2 (thin glass 1mm) as showed in table (2.1) and (2.2).

Table 2.1. *Composition of thick glass (4mm):*

Component	**Percent by weight**
SiO_2	*74.25%*
Na_2O	*13.04%*
CaO	*7.07%*
MgO	*3.98%*
Al_2O_3	*1.08%*
K_2O	*0.08%*
TiO_2	*0.06%*

Table 2.2. *Composition of thin glass*(*1mm*)**:**

Component	Percent by weight
SiO_2	73.17%
Na_2O	14.56%
CaO	5.99%
MgO	3.4%
Al_2O_3	1.63%
Fe_2O_3	0.15%
K_2O	0.14%

Type (1) Thick glass (4mm); 112 slides from window sheets of soda lime glass. And type (2) thin glass (1mm); 42 already made (slides of microscopes) slides from soda lime glass used to conduct this study.

2.1.1.1. Cutting:

Cutting the thick glass to make suitable shapes of slides to conduct testing operations later where the size of slides was (100 mm length x 40 mm width and 4 mm thickness), (200 mm length x 200mm width x 4mm thickness to make Armor glass) for type (1) thick glass and (90 mm length x 25 mm width x 1 mm thickness) for type (2) thin glass. The procedure of cutting done by diamond.

2.1.1.2. Grinding of glass:

Chamfering and deburring, two terms used in the manufacturing of pieces, identifying methods for the finishing of machined pieces[53]. Chamfering involves having a bevel or edge, a groove or a furrow. Deburring is used to eliminate some rough corners or areas from the component after formation. While hand finishing is still popular, manufacturers switch to lower costs for automation and produce more predictable performance. The finishing process, which eliminates excess material remaining on the glass component after processing, is an essential stage. This means that the component fits within the framework of the standards, irrespective of whether its expected implementation is basic or extremely sophisticated. Deburring eliminates pieces of glass and imperfections that can conflict with the operation of the component. They can chip or break during service, destroy other parts and cause injury. Chamfering is widely used for symmetrical and smooth grooves in gear teeth and glass corners[53].

Hand finishing using a bench with a cutting tool or spindle grinder, in addition to brushing. Because the grinding and deburring are hand-made, they are expensive steps that makes the total cost of the work high. This will add obviously to the final price of a product. Hand finishing, although usually needed, can be inaccurate as well as inconsistent unless extreme precautions are taken; in addition, if the work piece do not meet characterization, reworking will be mandatory. Chamfering is considered to be one step in the process of finishing of glass [54]. Firstly, the edges must be chamfered before the surface of the work piece can be grounded to prevent edge damage. which can be done by placing the work piece on a silicon carbide disc and rotating the disc with a rocking motion. The rotating disc will thus wear off the sharp edge of the work piece (glass pieces). Chamfering should be done in 45-degree angle and done by grinding machine (see figure 2.1) with two levels of grinding (320 μm and 600 μm) respectively to get rid of sharp corner and release any microcracks on the corner to avoid fast crack when bending is applied (see figure 2.2). After chamfering, cleaning the slides by alcohol and wait until dry, to remove any remaining particles on the glass surface to be ready for coat processing.

Figure 2.1. *Grinding machine.*

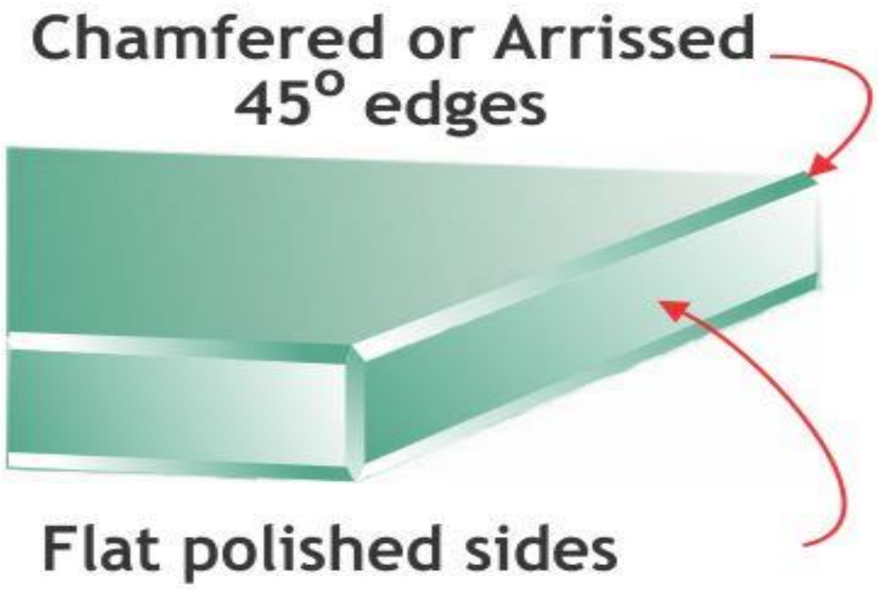

Figure 2.2. *Grinded glass slide.*

2.2. Paste preparation:

2.2.1. Materials*:*

Clay was liquified with 63.7% water and potassium nitrate powder in order to coat glass slides. Table (2.3) includes chemical composition of clay, Table (2.4) shows potassium nitrate properties. In this study clay was mixed with KNO_3 salt to make paste for coating the slides.

Table 2.3. *Chemical composition of clay percentages:*

SiO_2	Al_2O_3	CaO
11-14%	80-85%	3-4%

Table 2.4. *Potassium nitrate properties:*

Potassium Nitrate	: KNO_3
Molecular Weight/ Molar Mass	: 101.1032 g/mol
Density	: 2.109 g/cm^3
Boiling Point	: 400 °C
Melting Point	: 334 °C

2.2.2. Mixing:

Potassium nitrate salt mixed with (13.5 cm^3) of clay, the potassium nitrate salt calculated to be (63.7 %) of clay volume which equal to (24 cm^3 of KNO_3) in case, and (46.7 %) of clay which equal to (12 cm3 of KNO_3) in other case. In this study (KUNHEWHUA stirrer mixer model DLMYP2011-150, 60-1500 r/min used to mix clay with potassium salt (see figure 2.3). The clay mixed with salt for 15 minutes until getting homogeneous texture to be ready to coating prosses.

Figure 2.3. *Mixing equipment.*

2.2.3. Coating*:*

Coat or dip the slides of glass in paste carefully to ensure same thickness at surface for small slides then painting the remaining uncoated part of slides. While the large slides were coated all over the surfaces. Small slides, also, it can be coated not dipped in the mixed paste. The coated slides were kept in room temperature about one day or two days according to weather until drying. (See figure 2.4).

Figure 2.4. *coated glass slides.*

2.2.4. Tempering:

putting the dry slides in electrical Furance to make diffusion process on the surface of glass slides at 450 °C,500 °C,550 °C where the thin glass (1 mm) tempered only at 450°C because it come to viscosity at 500 °C. And other thick glass (4mm) tempered at 450 °C, 500 °C, 550 °C the melting point of potassium nitrate 334 °C, to ensure diffusion occurrence. The tempering process done by traditional electrical furnace (2.5) instead of special chemical tempering furnace that was used figure (2.6).

Figure 2.5. *Electrical Furnace.*

Figure 2.6. *Special chemical tempering furnace.*

- The tempering of glass slides classified according to 4 variables:

- Concentration of potassium nitrate in paste clay: The potassium nitrate mixed with 46.3 % volume of clay and 63.7 % of clay volume.
- Contact time: Three contact time (8,12,16) hours applied on glass slides to read the obtained results in each case.
- Temperature: Three degrees (450, 500, 550) °C of tempering temperature applied on each above cases. As showed in table (2.5).
- Thickness of slides glass: Two groups of glass thickness used in this study (1mm thin glass) and (4 mm thick glass).

Table 2.5. *The arrangement of groups according to variables of study.*

Groups No.	Thickness	Temperature	Concentration of KNO_3%	Time (h)		
1	4 mm	450 ^oC	46.7%	8 h.	12 h.	16 h.
	4 mm	450 ^oC	63.7%	8 h.	12 h.	16 h.
2	4 mm	500 ^oC	46.7%	8 h.	12 h.	16 h.
	4 mm	500 ^oC	63.7%	8 h.	12 h.	16 h.
3	4 mm	550 ^oC	46.7%	8 h.	12 h.	16 h.
4	1 mm	450 ^oC	46.7%	8 h.	12 h.	16 h.
	1 mm	450 ^oC	63.7 %	8 h.	12 h.	16

2.2.5. Cleaning:

clean the surface of tempered glass by tap water to remove the backed clay on glass surface, sometime need to use ultra sound instrument figure (2.7) for half hours to one hours to remove the remained dirts of backed clay (in case high temperature tempered like 550 °C), to be ready for testing. Figure (2.8) showing the difference between two tempered slides at 500 °C and 550 °C. The glass cleaned by ultrasound equipment model BANDELIN SONDREX - RK31H

Figure 2.7. *Ultrasonic bath.*

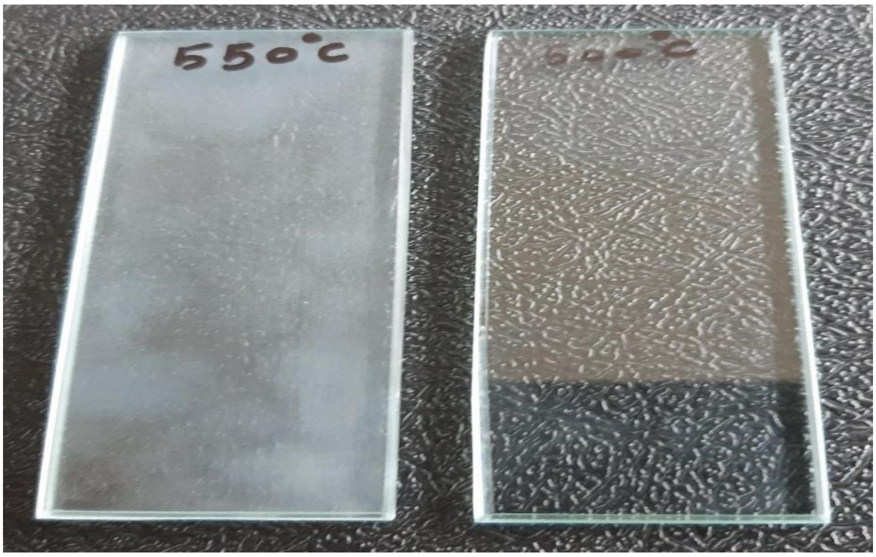

Figure 2.8. *The difference between tempered glass at 500C and 550C.*

2.2.6. Lamination of glass:

In this step 24 slides used to make Armor glass. 6 slides of thick tempered glass at 450 ^{o}C to 8, 16 hours with potassium nitrate concentration 46.7 %, 63.7 %. The slides dimensions were (200 mm x 200 mm) laminated with 5 PC polycarbonate adhesive sheets in between under negative pressure then heated to 140 ^{o}C for 1 hour. To be ready for ballistic impact test (See figure 2.9). The laminated glass classified to 4 groups. Group (A) tempered glass with 8 hours for 46.7 % potassium nitrate coat, group (B) tempered glass with 8 hours for 63.7 % potassium nitrate coat, group (C) tempered glass with 16 hours for 46.7 % potassium nitrate coat and group (D) tempered glass with 16 hours for 63.7 % potassium nitrate coat. The final size was (200 mm x 200 mm x 25 mm).

Figure 2.9. *Laminated glass.*

2.3. Characterization:

several types of destructive and non-destructive inspections used to test the strengthen glass slides. The bending test used to calculate the flexure strength of glass, hardness (H_V) test used to calculate the hardness of glass surface, SEM and EDX used to calculate the depth of potassium ion that obtained in the surface due to the diffusion process, and the other tests done to determine the changes in Optical properties like FTIR and surface residual stresses test also microscopy test used to determine crack behaviour. The results of each category compared with other researchers. The samples classified according to variables of glass: thickness of glass, contact time, concentration of potassium nitrate in clay, and tempering temperature, where group (1) for thick glass at 450 ^oC, group (2) for thick glass at 500 ^oC, group (3) for thick glass at 550 ^oC, group (4) for thin glass at 450 ^oC.

2.3.1. Bending test (3-points test):

Strengths of materials are usually reported in terms of the three-point bend strength or the flexural strength, frequently called the modulus of rupture (MOR). The stress at fracture using this flexure test is known as the flexural strength, modulus of rupture, fracture strength, or bend strength, an important mechanical parameter for brittle ceramics. For a rectangular cross section, the flexural strengths are equal to equation:(2.1), (2.2) [55].

$$\sigma_f(\mathrm{MOR}) = \frac{3\,F\,L}{2\,b\,d^2} \qquad \text{(for rectangular)} \qquad (2.1)$$

$$\sigma_f(\mathrm{MOR}) = \frac{F\,L}{\pi\,R^3} \qquad \text{(for circular)} \qquad (2.2)$$

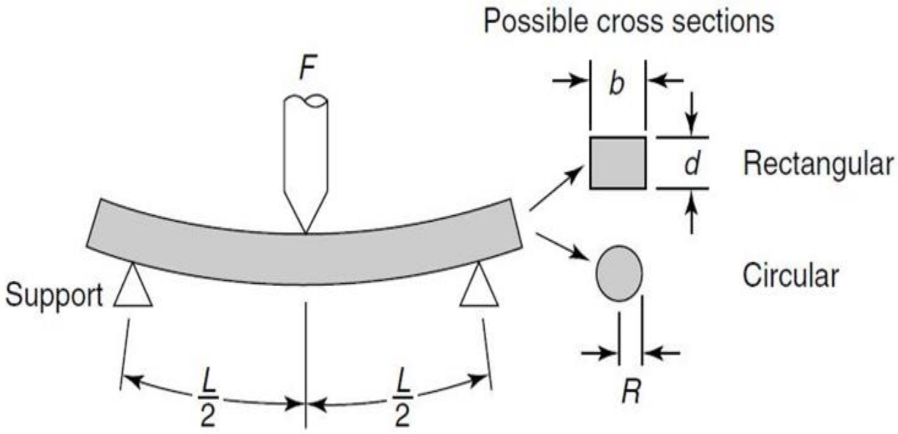

Figure 2.10. *Sketch represented 3-point test [39].*

Where: σ_f is the strength in three-point bending, (**L**) is the length of the test span, (**b**) is the specimen width, (**d)** is the specimen height, the fracture load is (**F)**, when the cross section is circular and (**R)** is the specimen radius. The flexural strength will depend on sample size, since with increasing size there is an increase in the probability of the existence of a crack-producing flaw, along with a corresponding decrease in the flexural strength. This category of bend flexures is designed for 3-point and 4-point bend testing of plastics, metals, ceramics, tubing and many other materials. Two base sizes are available, along with an upper anvil, and optional roller diameters. Use a base-anvil combination for 3-point bend testing (figure 2.10) or two bases for 4-point bend testing. The flexure test was according to ASTM C393, C1161, D790, and related standards [55]. In this test 96 samples (100 mm x 40mm x 4mm) size of thick glass, and 42 slides (75mm x 25mm x 1mm) size of thin glass tested, in each category 6 samples, lowest and highest value were neglected in flexure strength data. The bending test is done by INSTRON model (5581) machine, see figure (2.11). Using load rate 0.5 N/sec.

Figure 2.11. *Instron bending test equipment.*

2.3.2. Hardness test (H_V):

Two common hardness-testing methods are Knoop and Vickers (usually referred to as a diamond pyramid). For every test, a precisely small diamond indenter of pyramidal configuration is pushed into the surface of the test piece. The applied loads are very much less than those of Brinell and Rockwell, varying from 1 to 1000 g [55]. The resultant effect is examined and measured under a microscope; this value is then transformed to a number of hardness. Careful treatment of the surface of the specimen (chamfering and polishing) might be required to ensure a precise indentation that can be accurately determined. The Vickers and Knoop hardness numbers are H_V and H_K, respectively. The hardness measurements for both methods are roughly equal. Vickers and Knoop are related to as micro indentation-testing approaches focused upon indenter thickness. These are well adapted for evaluating the toughness of thin specimen slides regions; Knoop is often applied for checking fragile materials such as glass and ceramics. The new micro-indentation system of hardness-testing has become simplified by linking the indenter unit to a computer and software kit with an image analyser. Code monitors critical device functions that include indent position, indent spacing, hardness value computation, and data analysis. In this study Vickers hardness was used to determine hardness of strengthened glass by microhardness machine showing in figure (2.12). Model (M1C 010) with 621.6 x lens and 1 kg load at 10 sec total time according to ASTM Standard E-384, "Standard Test Method for Micro Indentation Hardness of Materials.". One slide from each category randomly selected to determine hardness of glass, where the slides tested for 10 times in micro indentation, the highest and lowest readings were neglected to determine mean of hardness.

Figure 2.12. *Microhardness device.*

2.3.3. Microscope test:

Microscope is a tool that is used to magnify the things (picture), in order to visualize the specimen in a microscopic level, so they can be tested appropriately. Usually it's used in medical and scientific laboratories. In this test (Zeiss microscopes model primo star was used to inspect crack behavior in the glass slides) figure (2.13).

Figure 2.13. *Stereo-microscope instrument.*

2.3.4. SEM and EDX analysis:

The scanning electron microscope (SEM) permits the observation and characterization of heterogeneous organic and inorganic materials on a nanometer (nm) to micrometer (μm) scale. The popularity of the SEM due to its capability of obtaining three-dimensional-like images of the surfaces of a very wide range of materials. SEM images are used in a wide variety of media in scientific journals. Although the major use of the SEM is to obtain topographic images in the magnification range 10–10,000×, the SEM is much more precise. In the SEM, the area to be examined or the microscale to be analyzed is irradiated with a finely focused electron beam, which may be swept in a raster across the surface of the specimen to form images or may be static to obtain an analysis at one position. The types of signals produced from the interaction of the electron beam with the sample induce secondary electrons, backscattered electrons, x-rays and other photons of various energies. These signals are obtained from specific emission electron within the sample and can be used to examine many characteristics of the sample (surface topography, crystallography, composition, etc.). The imaging signals of greatest interest are the secondary and backscattered electrons because these vary primarily as a result of differences in surface topography. The secondary electron emission, confined to a very small volume near the beam impact area for certain choices of the beam energy, permits images to be obtained at a resolution approximating the size of the focused electron beam. The three-dimensional appearance of the images is due to the large depth of field of the scanning electron microscope as well as to the shadow relief effect of the secondary and backscattered electron contrast [56].

In this test (SEM Zeiss Supra 40VP - figure 2.14) was used to calculate depth of potassium ion in glass. The main variable to study was tempering temperature, other variables were taken but gave similar results, for this reason were not mentioned in details.

Figure 2.14. *SEM instrument.*

2.3.5. Surface residual stress test:

Residual stresses in tempered glass are generated by interactions between thermal contraction, elasticity at low temperatures, viscoelastic flow at higher temperatures, and temperature gradients caused by cooling**.**

Surface residual stresses done by visual test. Where a beam of white light focused on surface of glass for a known distance to see the reflected light. Where in case of disturbed optical properties, the focused beam white light scattered to a spectrum of visible light. In case of non-disturbed optical properties will not be scattered to spectrum of light as shown in figure (2.15), commpared with glass slide tempered by thermal method (disturbed optical properities) showing in figure (2.16).

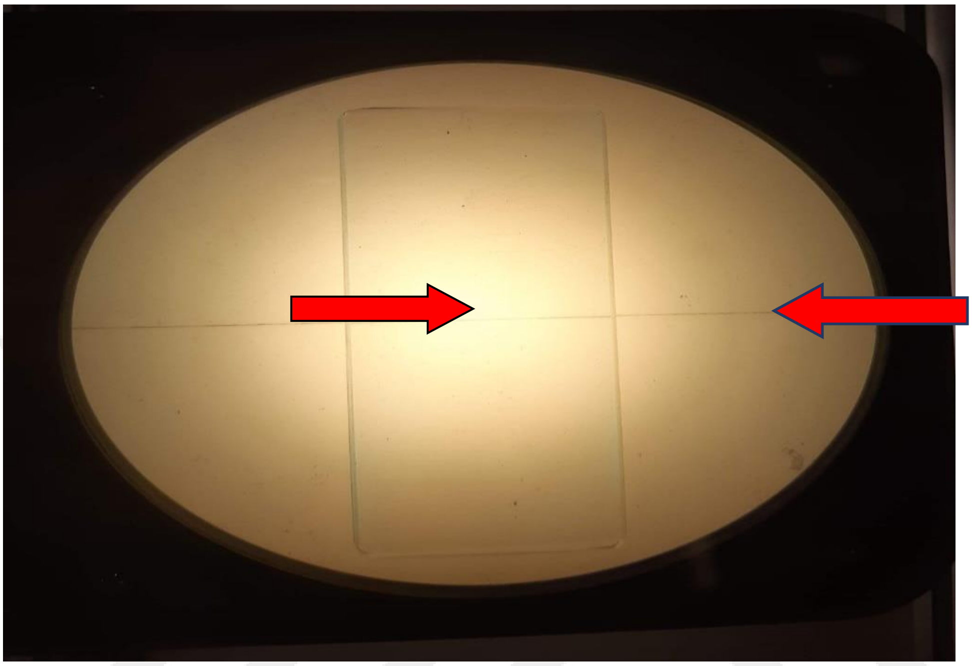

Figure 2.15. *Chemical tempered glass (new method).*

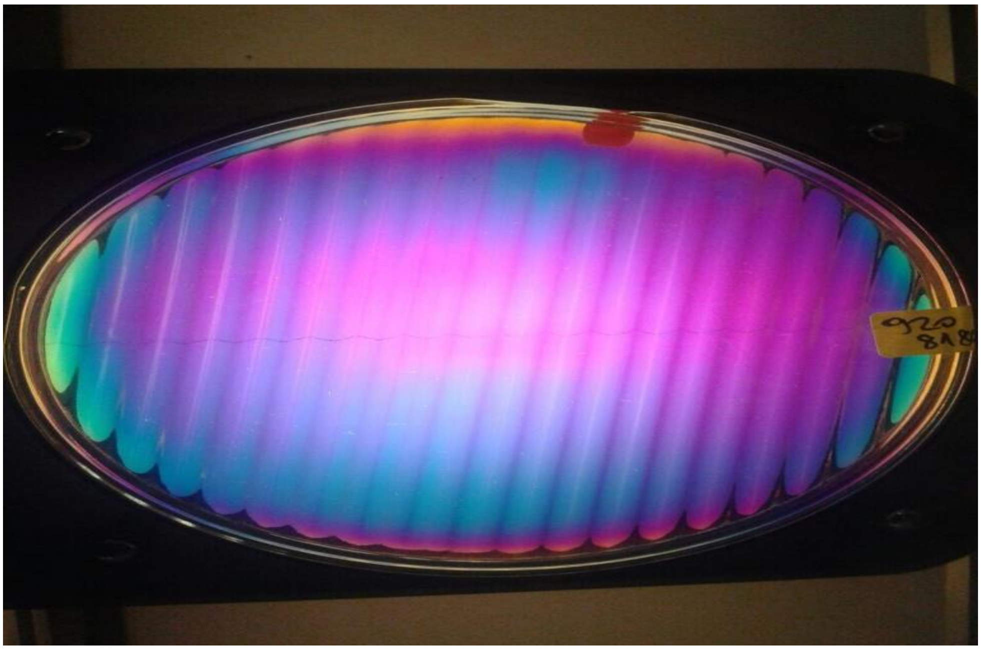

Figure 2.16. *Thermal tempered glass.*

2.3.6. Fourier Transform Infrared Spectroscopy (FTIR):

The instrument that calculate the absorption spectrum for a material is called a spectrophotometer. Fourier transform infrared spectrophotometer provides the IR spectrum rapidly compared to the traditional spectrophotometer. The instrument produces a beam of IR irradiation, which is emitted from a glowing black-body source. Subsequently, the beam passes through interferometer where the spectral encoding takes place. The recombination of beams with different wave lengths in the interferometer creates constructive and destructive interference called an interferogram [57]. In the meantime, a beam is superimposed to provide a reference (background) for the operation. Finally, the desirable spectrum was obtained after the interferogram automatically, subtracted the spectrum of the background from the sample spectrum by Fourier transformation computer software. Basically, the IR spectrum obtained from FTIR spectrometer lies in the mid-IR region 2.5–15 μm between 4000 and 666 cm^{-1}. Transition energies corresponding to changes in vibrational energy state for many functional groups are located in the mid-IR region (4000–400 cm^{-1}) and hence the appearance of an absorption band in this region can be used to determine whether specific functional groups exist within the molecule [57]. The FTIR instrument used to determine transmittance of tempered glass. The test done by BRUKER model tensor 27 (see figure 2.17), with wave length until 4000 cm^{-1}. One slide of each category randomly selected to tested by FTIR.

Figure 2.17. *FTIR instrument.*

2.3.7. Ballistic impact test:

Ballistics can be defined as “the scientific study of material resistance which is shooted or fired through the air, with shots such as bullets and missiles” [58]. The military industry is divided in two aspects: research and development of weapons – guns, rifles, cannons, and development of ballistic protection – shields, body and vehicle armor, and helmets [59]. The development of ballistic protection materials is conducted based on two main factors: what is to be protected and the desired protection level [60]. The test was done with launcher machine (see figure 2.18) according to European standard EN-STD-1063-BR6 for a 7.62 bullet incident with speed 895 m/s and EN-STD-1063-BR2 for a 9 mm parabellum bullet fired with a speed of 373 +_ 9.1 m/s 8 g. and the aim of this study was to suggest a laminate design that would increase the ballistic resistance of bulletproof materials of tempered glass [61].

NP = No Penetration - The projectile did not completely penetrate the test specimen.

CP = Complete Penetration - The projectile completely penetrated the test specimen.

PP = Partial Penetration - A situation when the projectile completely penetrates a test specimen, but projectile itself did not exit the specimen. The projectile is lodged and protruding out the rear surface of the specimen.

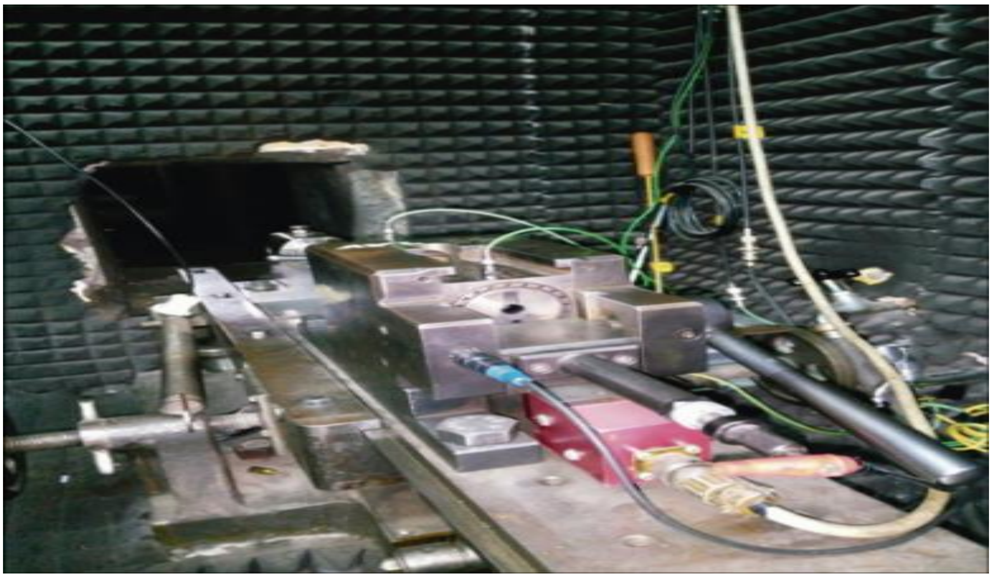

Figure 2.18. *Ballistic impact test equipment.*

3. RESULTS AND DISCUSSION:

The results of all tests were classified according to the mentioned categories, while the contact time kept as the main variable for comparisim. The glass tempered with three temperatures (450, 500, 550 °C) except for thin glass in which the slides were melt down in 500 ^{o}C. For that reason, the thin glass was tempered at only 450 °C.

3.1. Effect of Temperature:

The thick tempered glass with 450 ^{o}C (group 1) recorded increasing flexural strength up to (195 MPa) 3.20 folds in best reading, the group (2) 500 ^{o}C tempered glass resulted increasing flexural strength up to (290 MPa) 4.75 folds in best reading, where the 550 ^{o}C (group 3) recorded increasing flexural strength (84 MPa) 1.4 folds as a best record. The readings of tempered glass compared with untreated glass that has flexural strength (61 MPa). Figure (3.1) showed cyclical pattern with contact time, where increase tempering time leads to decrease strength that was clearly in case of group (2) 500 ^{o}C tempered glass. While in case of 450 ^{o}C increasing tempering time to 16 hours recorded slightly increased strength comparing with 8 hours, this may be due to relaxation and reversible reaction explain cyclical pattern.

Kim H.J et-al. recorded four folds increment of glass strength although used different glass [62].

This disagree with Erdem I. et-al who mentioned decrease in glass strength with increasing temperature and increased with increasing contact time [63]. and agrees with Rene Gy that shows the increasing of temperature leads to increase strength of glass [64],

Varshneya A. and Sglavo V.M. mentioned increasing contact time leads to decrease strengthening due to relaxation [27][65].

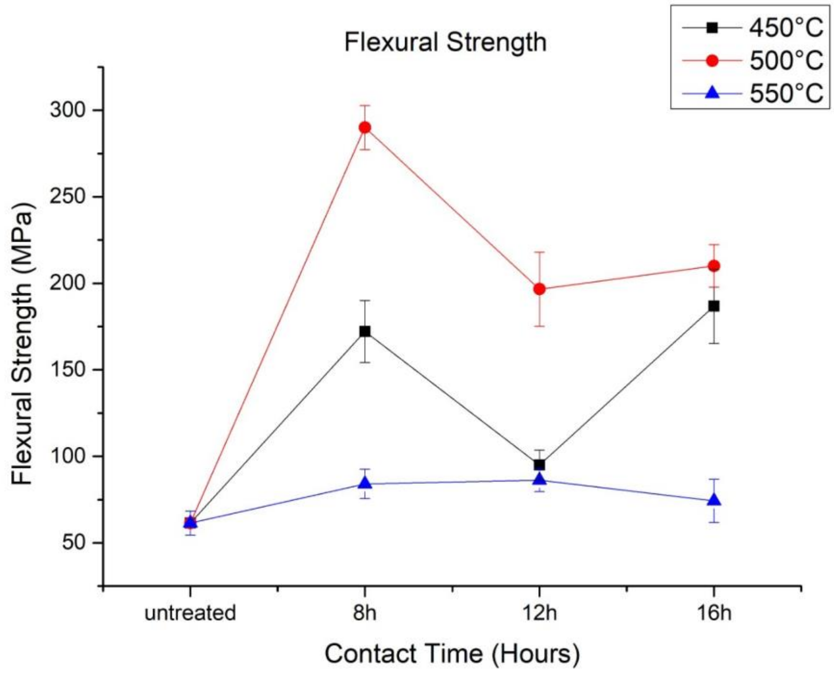

Figure 3.1. *Tempering temperature effect on flexural strength of thick glass.*

The microscope test showed zig zag layer at tempered glass surface, Where the untreated glass did not show zig-zag behaviour (see figure 3.2). The tempered glass has an un-uniform zig-zag layer changes the path of crack, this layer reaching up to 400-500 μm depth in case of 450 °C tempered thick glass (4mm) figure (2.3), in case of 500 °C tempered glass showed un-uniform clear zig-zag layer 1500-2000 μm depth (figure 2.4), and 600 μm depth slightly zig-zag layer in case of 550 °C (see figure 2.5).

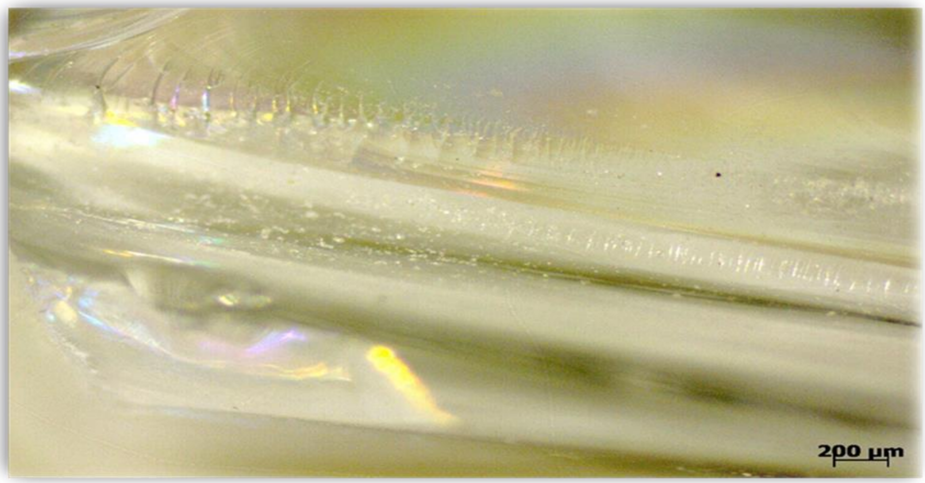

Figure 3.2. *Cross section of untreated glass.*

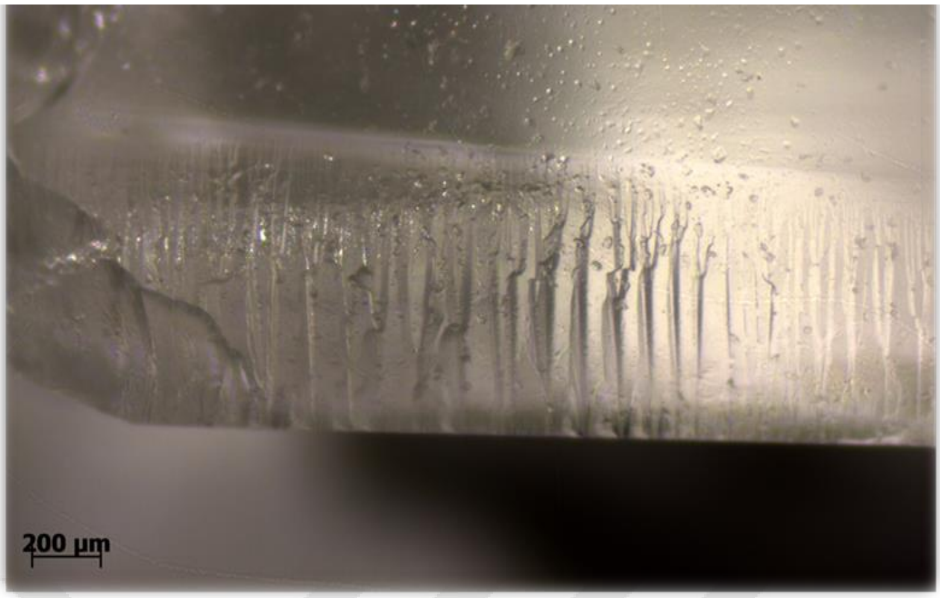

Figure 3.3. *Cross section represents crack behaviour for 450 ^{o}C tempered glass.*

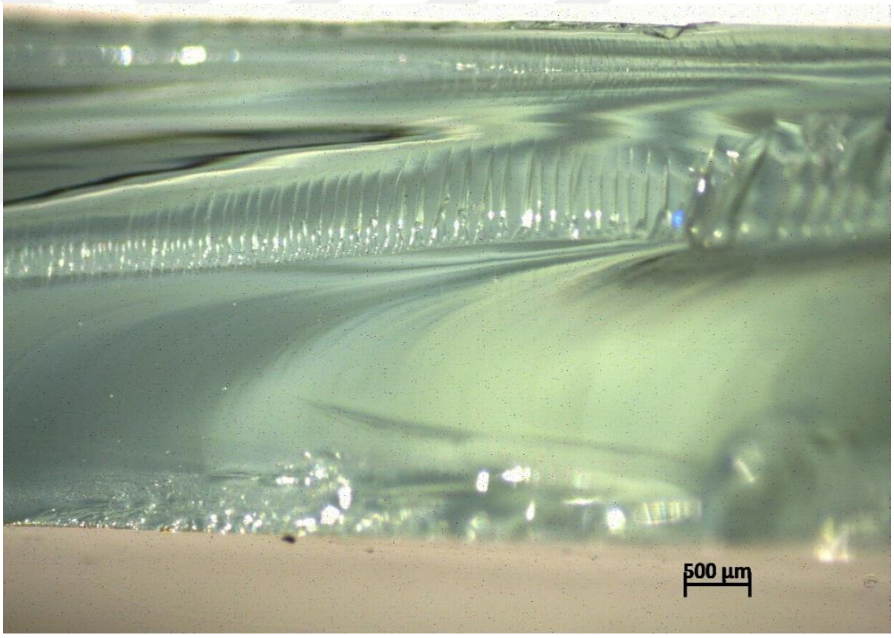

Figure 3.4. *Cross section represents crack behaviour for 500 ^{o}C tempered glass.*

Figure 3.5. *Cross section represents crack behaviour for 550 °C tempered glass.*

In the EDX analysis there was clear correlation between the temperature and the depth of potassium ion exchange in each tempering temperature see figures (3.6), (3.7) and (3.8). This explains the improvement of mechanical properties with temperature except for 550 °C group (3), where the increasing of potassium ion exchange depth did not improve the mechanical properties where diffused other elements of clay because of 550 °C was so close from vitreous transition degree of glass (570 °C). That was clear in figure (2.8). Table (3.1) showing the effect of potassium ion depth on flexural strength of glass.

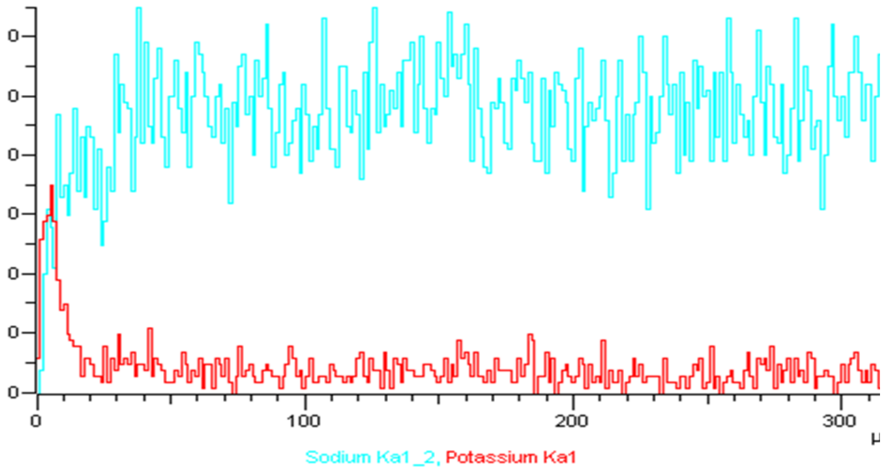

Figure 3.6. *EDX analysis of potassium ion depth in tempered glass at 450 °C.*

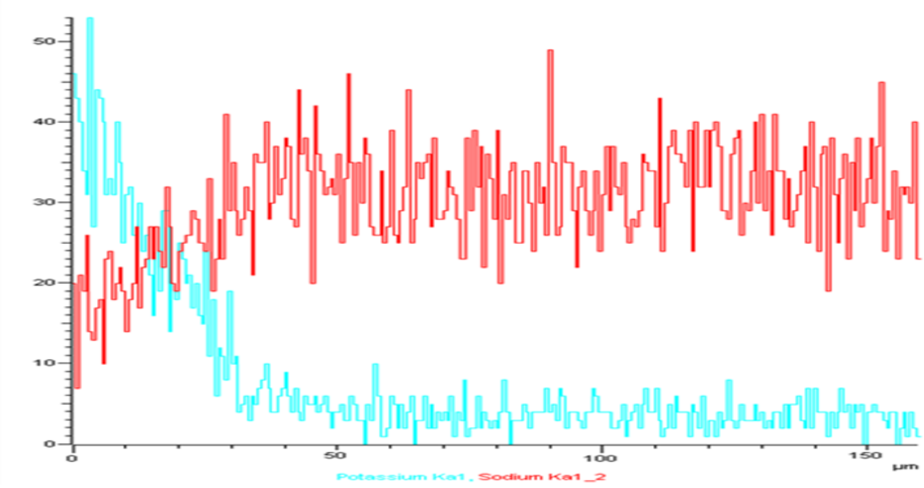

Figure 3.7. *EDX analysis of potassium ion depth in tempered glass at 500 °C.*

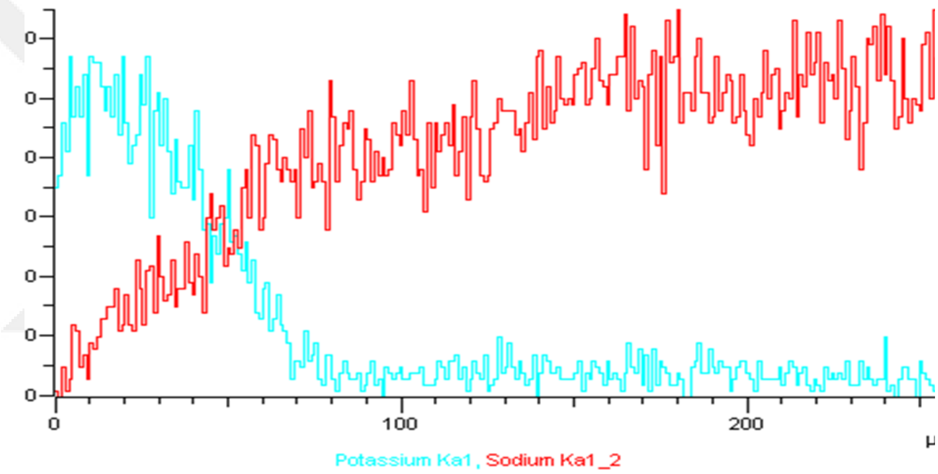

Figure 3.8. *EDX analysis potassium ion depth in tempered glass at 550 °C.*

Table 3.1. *Effect of tempering temperature on mechanical properties of glass and depth of potassium ion.*

Temperature	Flexure strength (MPa)	Depth (μm).
Untreated glass	61	#
450 °C	195	15
500 °C	290	30
550 °C	84	70

3.2. Effect of Salt Concentration (KNO_3):

The increasing salt concentration in glass coating recorded increase in flexure strength of tempered glass comparing in same tempering temperature. Where the 46.7% salt concentration recorded (172, 94, 186) MPa for (8, 12, 16) hours respectively, and 63.7% salt concentration recorded (195, 137, 195) MPa for (8, 12, 16) hours respectively. Also increasing in contact time leads to cyclical pattern (figure 3.9) where the flexure strength decreases in case of 12 hours, while 16 hours resulted in slightly increasing strength compared with 8 hours tempered glass at 450 °C. This cyclical pattern may be due to reversible reaction between diffused ions as explained above.

Sglavo V.M. took different salt purities for ion exchange and shows revers relation between purity and flexural strength of glass [65].

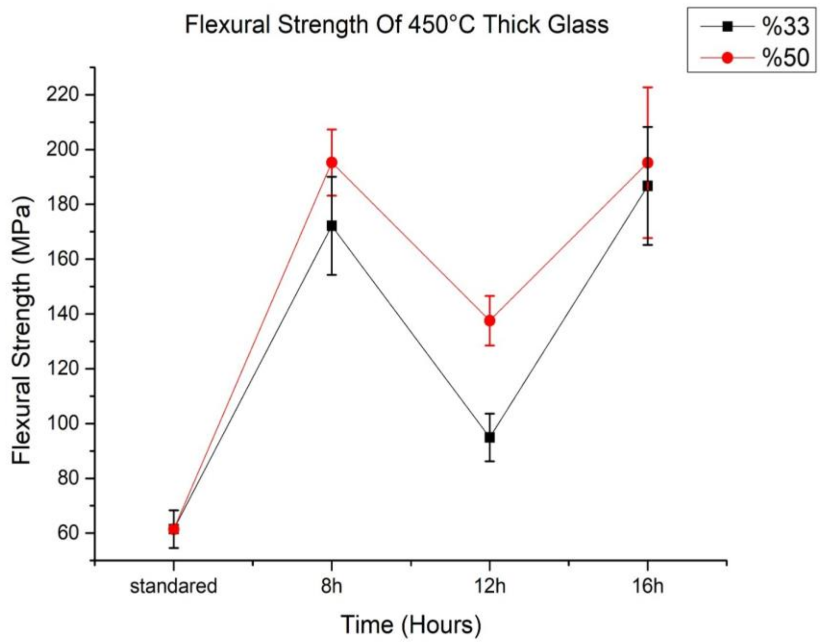

Figure 3.9. *Salt concentration effect on flexural strength of thick glass,*

EDX test showed same depth of potassium ion approximately and higher intensity in same tempering temperature and contact time, that explain increasing strength of tempered glass when increase concentration of salt. Which clearly showing in Figure (3.10), (3.11) for 500 °C tempered glass at 16 hours. Table (3.2) explain effect of increment salt concentration on strength of tempered glass. This result agreeing with Erdem I. et-al, Rene Gy, [63], [64].

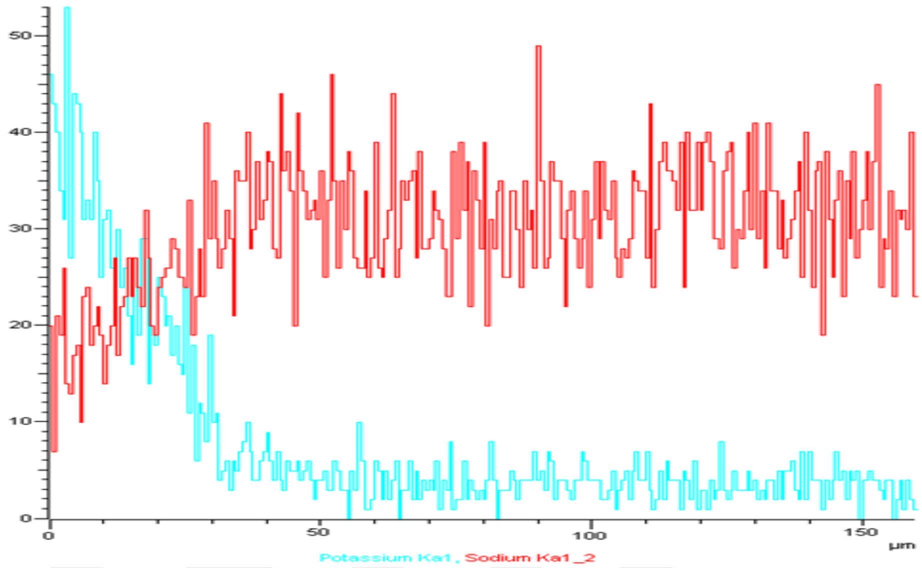

Figure 3.10. *ESD analysis of Potassium ion intensity at surface in case of 63.7%.*

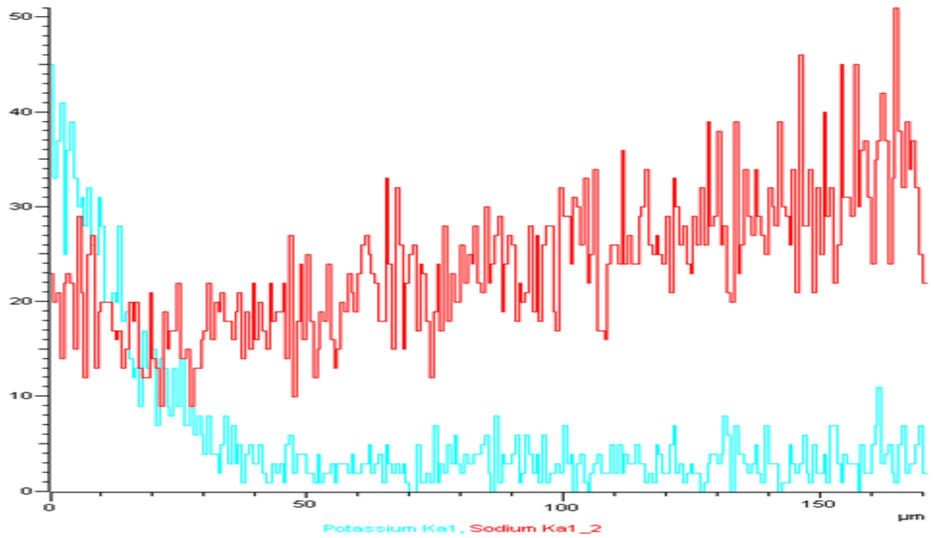

Figure 3.11. *EDX analysis of Potassium ion intensity at surface in case of 46.7%.*

Table 3.2. *Effect of increment salt concentration at 450 °C.*

Time / C %	Flexural strength (MPa)	Depth (μm)	Intensity
8	23 increment	15	↑
12	41 increment	15	↑↑
16	9 increment	15	↓

3.3. Effect of Glass Thicknesses:

The thickness of the glass effected the tempering process inversely, so that the result of thin glass has been better than thick glass readings, nevertheless, the limitation of thin glass tempering it cannot be treated at 500 °C. because the glass melts down. The thin glass (1mm) recorded (241MPa) in a best mean reading which means increasing strength about 3.59 folds at 450 °C. While the thick glass (4mm) recorded (195 MPa) in a best mean reading that refers to increasing strength to 3.19 folds in same tempering temperature.

The ratio of ions penetration depth to glass thickness is the determinant of the strength increment between them.

$$\mathbf{F.\ S.} \sim \frac{Pentretion\ depth}{Thickness} \tag{3.1}$$

Where thin tempered glass has increasing of strength about 12.5 % comparing with thick tempered glass (see figure 3.12). The ratio between ions depth layer to thickness of glass is responsible of this increment. where the EDX analysis recorded (25 $\boldsymbol{\mu}$m) potassium ion depth for thin glass (see figure 3.13), while thick glass recorded (15 $\boldsymbol{\mu}$m) depth as showed in figure (3.6) above.

Kim H.J rt-al 2019. recorded four folds increment of glass strength although used different glass [62]. Erdem et-al 2017 was able to treat 1 mm thick slides in 500 °C because the usage different (SLS) chemical composition with 2.82 folds at 16 hours [63]. Macrell.G et-al investigated 11 samples of 1mm thickness of (SLS), obtained 255- 316 MPa compared with 57-91 MPa for annealed glass [66].

Rene Gy 2008 obtained 250 Mpa. For 3 mm of (SLS) compared to 290 MPa. In case of 4 mm [64]. Sglavo V.M. obtained 593 MPa (2.3) folds in chemical strengthening to 4 mm thickness [65].

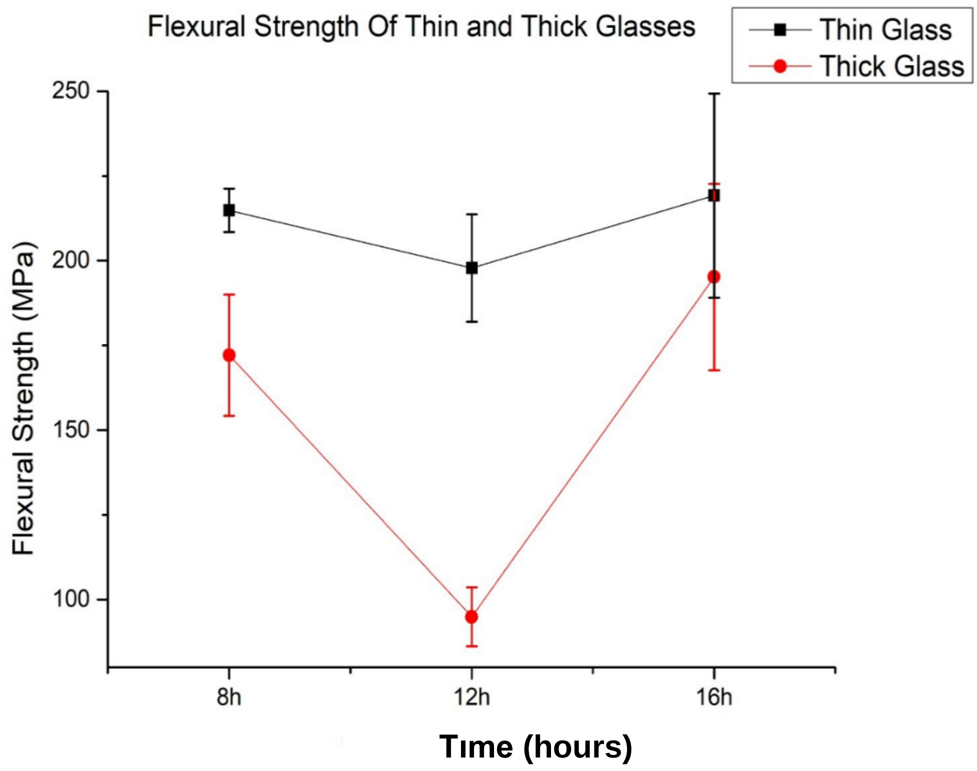

Figure 3.12. *Thickness effect on flexural strength of thin and thick glass.*

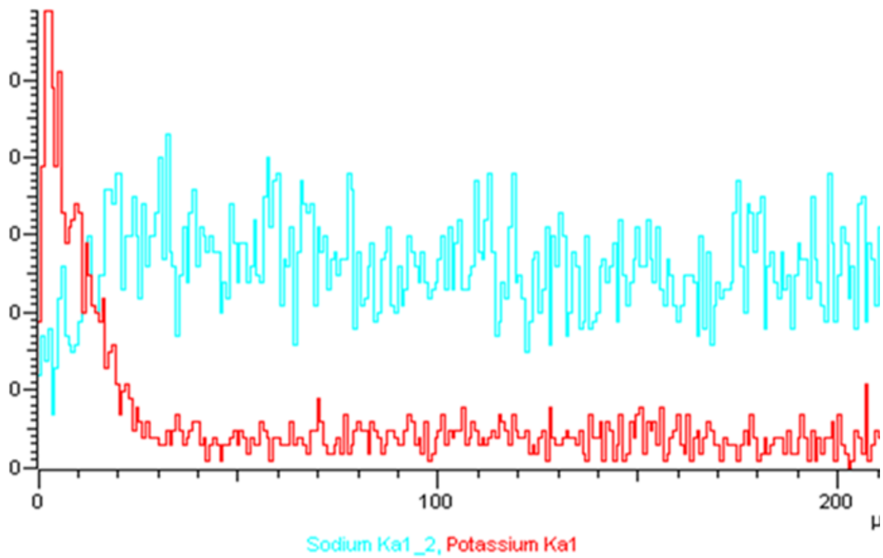

Figure 3.13. *EDX analysis of potassium ion depth in thin glass (1 mm).*

The crack behaviour of thin glass showed uniform zig-zag layer at glass surface along 160 μm (figure 3.14) which refers to homogeneity of ion penetration in glass, while thick glass showed un-uniform zig-zag layer (see figure 3.3 in above).

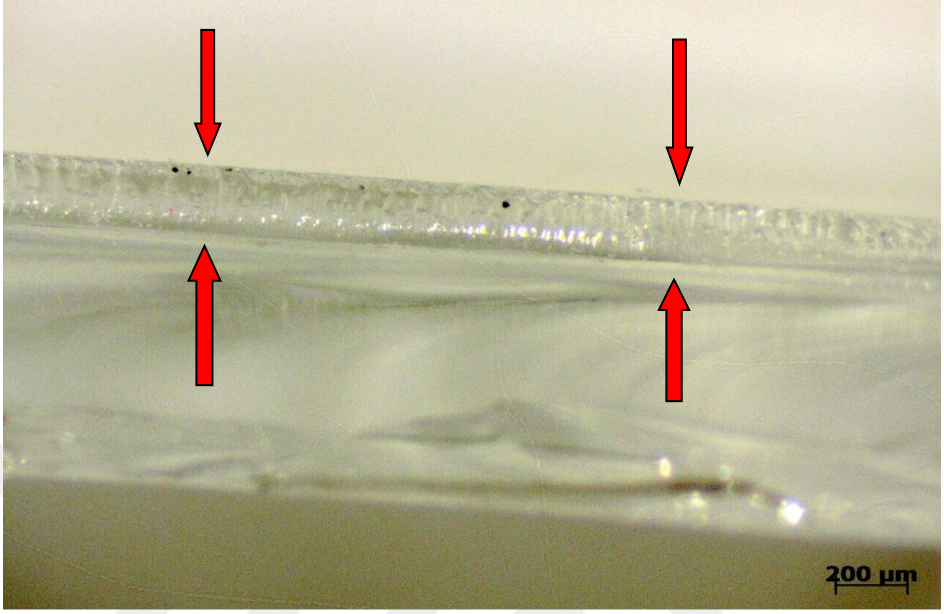

Figure 3.14. *Cross section represents crack behaviour of thin glass (1 mm).*

3.4. Effect of Chemical tempering on Hardness of glass:

Chemical tempering showed increment about (9%, 14%, 12 %) in a best mean result of hardness for (450, 500, 550) ^{o}C tempered glass respectively showing in figure (3.15), except in 12 hours for 550 ^{o}C recorded a slight decrease, While the hardness of thin tempered glass resulted about 15 % increment. Figure (3.16) showing hardness of thin tempered glass.

Erdem et-al. agrees with ascending pattern taking inconsideratiOn that their study was on 1mm thickness only [63].

Hwan Kim D. et-al. mentioned a hardness of 520 H_V in classical method of chemical tempering [67].

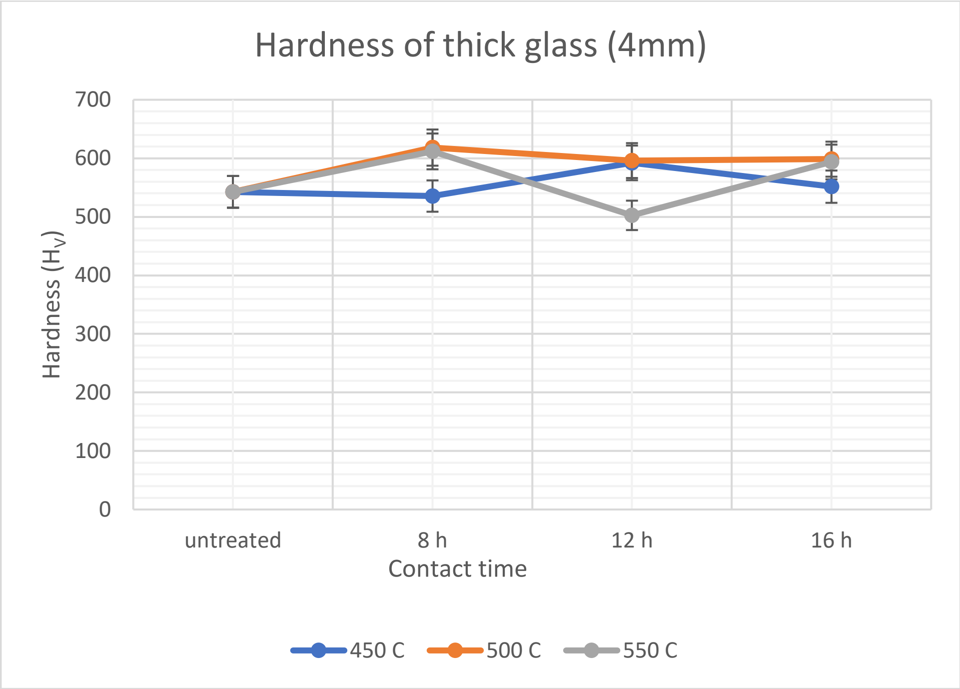

Figure 3.15. *Hardness of thick tempered glass(4mm) for (450, 500, 550 ºC).*

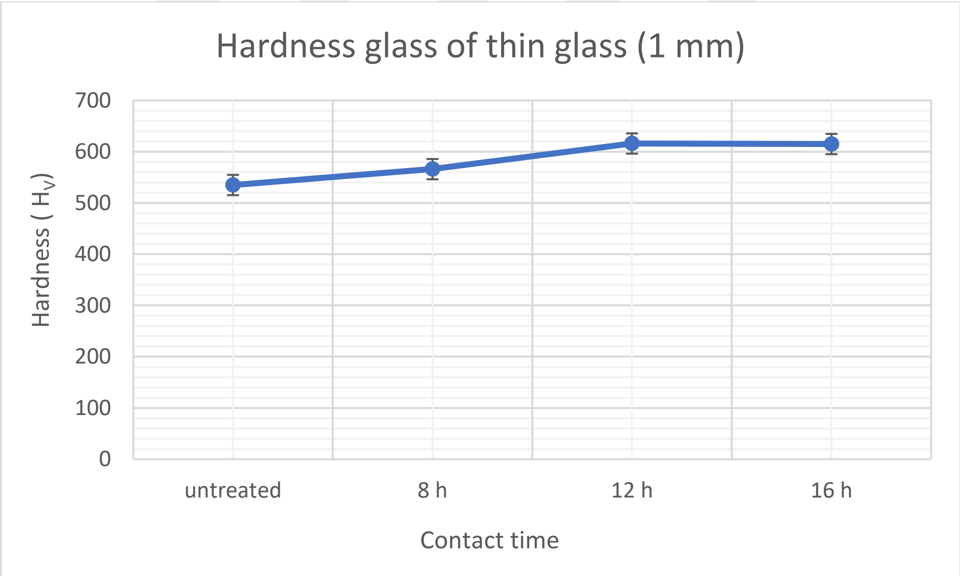

Figure 3.16. *Hardness of thin tempered glass(4mm) for (450 ºC).*

3.5. Effect of chemical tempering on Optical properties:

The transmittance property showed slight improvement for 450ºC tempered thin glass (1 mm) see figure (3.17), and unaffected for thick tempered glass (4mm) for 450ºC as showing in figure (3.18).

Increasing in contact time leads to increase transmittance of glass in case of thin glass at 450 °C, and decrease transmittance with increase contact time in case of high temperature.

Decreased transmittance of 500 °C and 550 °C see figure (3.19, 3.20). Although visual inspection of the tempered glass slides of 450 °C, 500 °C showed good translucency while 550 °C showed white opacities may be due to unwanted elements interdiffusion because the temperature was close to vitreous transition degree. Maeng J.H. mentioned decreased transmittance of (SLS) taking in consideration he used etching method [68].

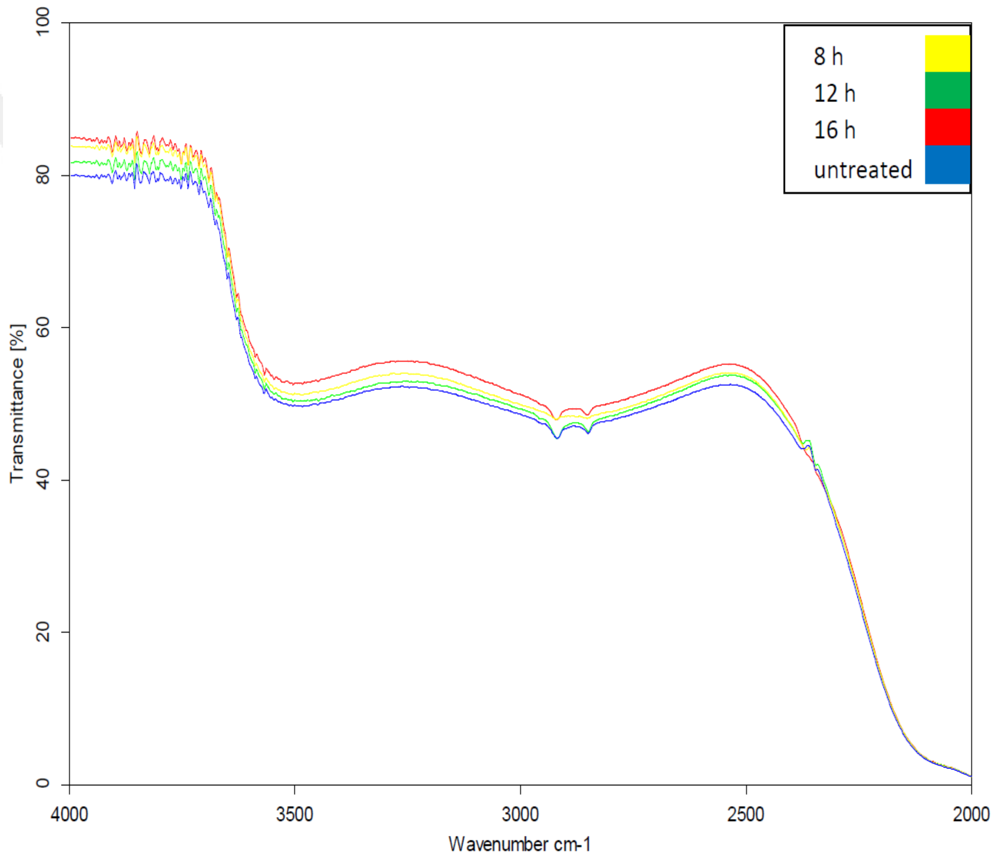

Figure 3.17. *Transmittance of thin glass(1mm).*

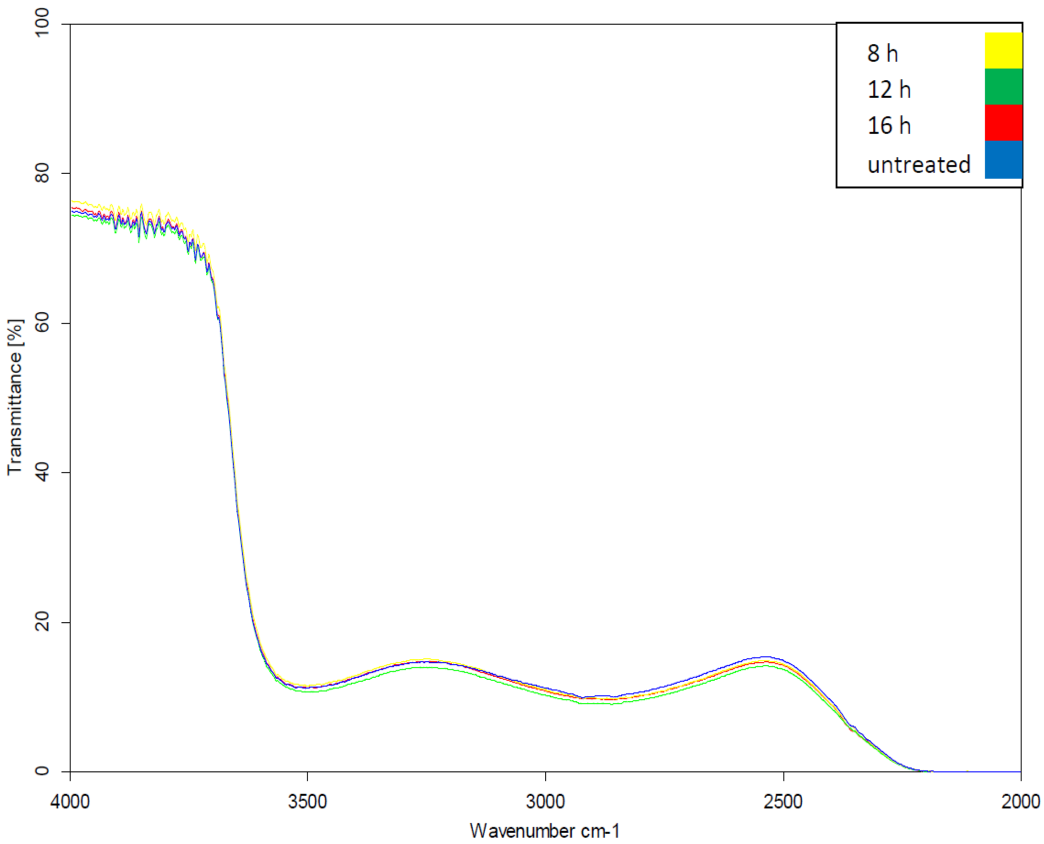

Figure 3.18. *Transmittance of thick glass (4 mm) at 450 ºC.*

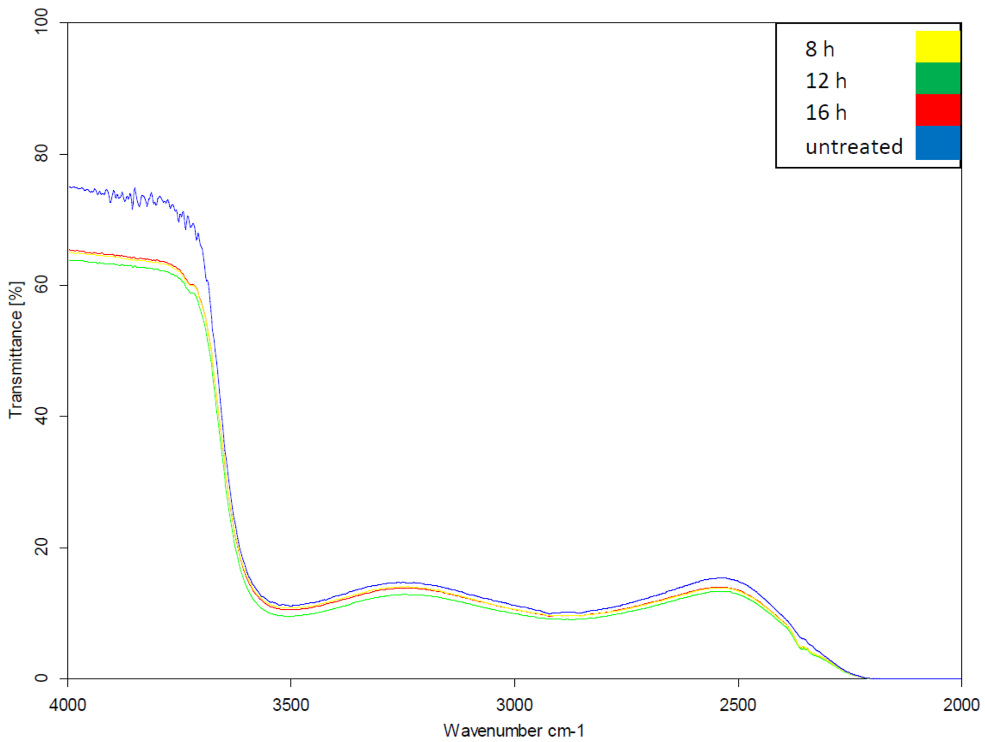

Figure 3.19. *Transmittance of thick glass (4 mm) at 500 ºC.*

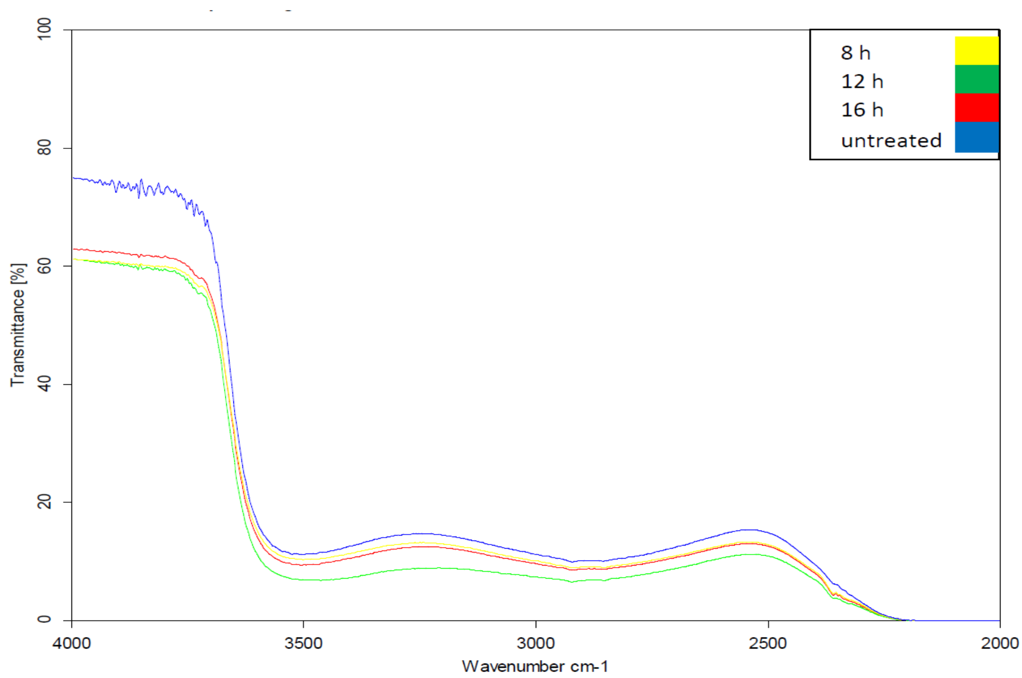

Figure 3.20. *Transmittance of thick glass (4 mm) at 550 °C.*

3.6. Ballistic Impact Test Results:

Four Armors glass tested with EN1063 standard for level 2 and one of this four Armors glass (group C) tested by level 6.

Group (A): 450 °C-8 h.-46.7 % potassium nitrate coated Armor glass tested by level 2 with same conditions and resulted partial penetration PP for 5 shooting. And one shoot results bulging because the shoot was about 5 mm around 3, 4 shoots so that rested completely penetrated CP. table (3.3) include tests conditions of 450-8h.-46.7 % potassium nitrate coated Armor. Figure (3.21) and (3.22) shown front side and back side after impact test.

Table 3.3. *Level 2 impact test conditions of Armor group (A).*

No. of shots	Type of bullet mm	Ballistic standard and striking distance	Speed (m/s)	Mass (g)	result
1	9 x19	EN1063-BR2	392	8±1	PP
2	9 x19	EN1063-BR2	395	8±1	PP
3	9 x19	EN1063-BR2	388	8±1	PP
4	9 x19	EN1063-BR2	376	8±1	CP
5	9 x19	EN1063-BR2	384	8±1	PP
6	9 x19	EN1063-BR2	382	8±1	PP

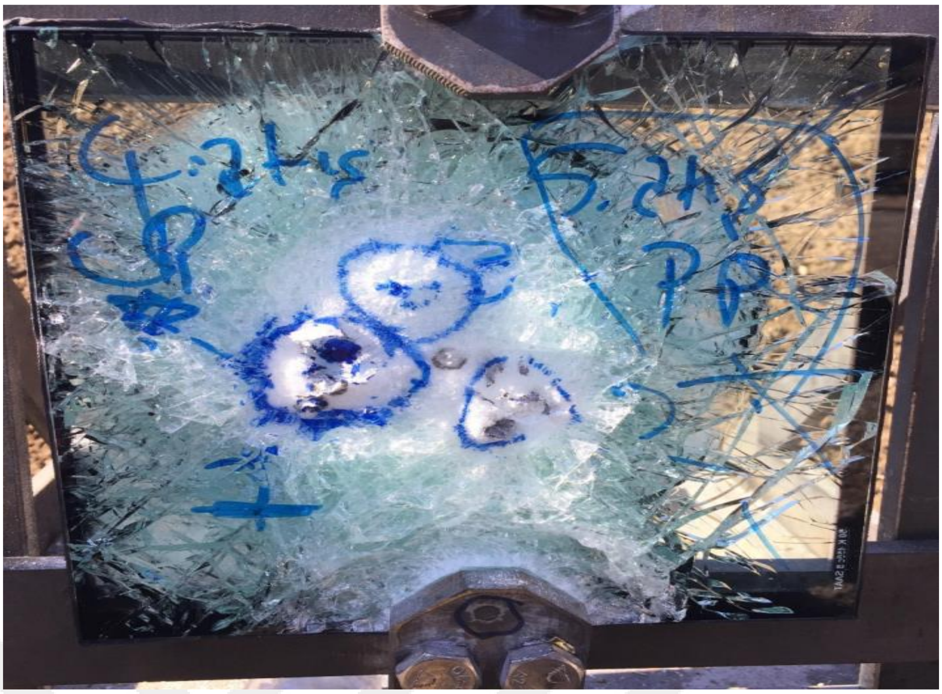

Figure 3.21. *Group (A) armour glass front side after impact test.*

Figure 3.22. *Group (A) armour glass back side after impact test.*

Group (B): 450C-8h.- 63.7% potassium nitrate coated Armor glass. 5 shoots resulted partial penetrated for all shoots and same conditions test. Table (3.4) shown result and figure (3.23) and (3.24) shown front and back side of armor glass after impact test.

Multi shot performance can be evaluated that the test sample has high multi-shot performance and trauma behavior is higher than normal glass.

Table 3.4. *Level 2 impact test conditions of Armor group (B).*

No. of shots	Type of bullet (mm)	Ballistic standard and striking distance	Speed (m/s)	Mass (g)	result
1	9 x19	EN1063-BR2	392	8±1	PP
2	9 x19	EN1063-BR2	395	8±1	PP
3	9 x19	EN1063-BR2	388	8±1	PP
4	9 x19	EN1063-BR2	376	8±1	PP
5	9 x19	EN1063-BR2	384	8±1	PP

Figure 3.23. *Group (B) armour glass front side after impact test.*

Figure 3.24. *Group (B) armour glass back side after impact test.*

Group (C) :450 °C-16 h. 46.7% potassium nitrate Armor tested with level 6 by 9.5 g bullet ,815 m/s speed of bullet, and 120 mm striking distance for impact test. The result was failure and the bullet completely penetrated but not overall mass (figure3.28). Table (3.5) shown conditions of level 6 test, figure (3.25), (3.26), (3.27) shown glass armors group (3) before impact test and the damages of front side and crown shaped deformation in back side after impact test respectively.

Table 3.5. *Level 6 impact test conditions of Armor group (C).*

No. of shots	Type of bullet(mm)	Ballistic standard and striking distance	Speed (m/s)	Mass (g)	result
1	7.62 x51 mm (M80 ball)	EN1063-BR6	815	9.5±1	CP

Figure 3.25. *Group (C) armour glass before impact test.*

Figure 3.26. *Group (C) Armour glass after level 6 impact test.*

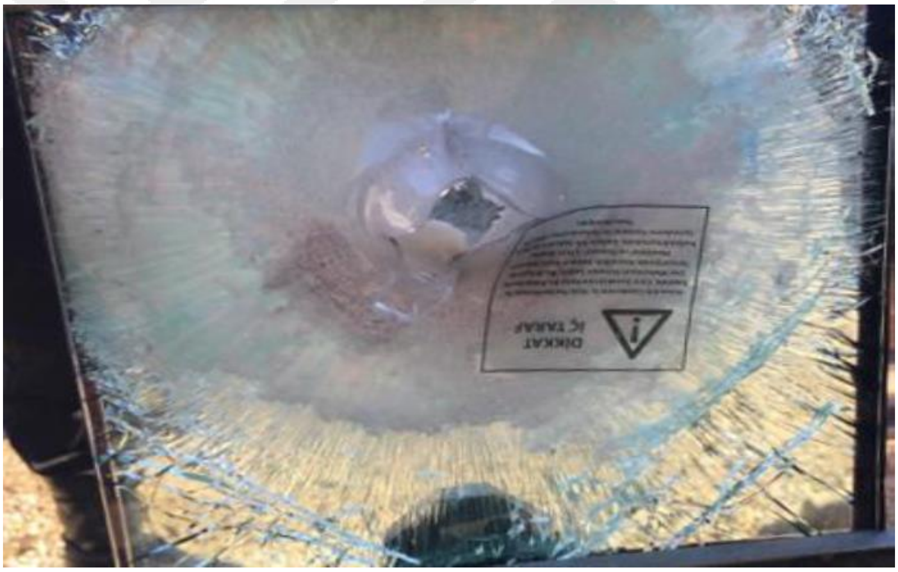

Figure 3.27. *Group (C) Armor glass back side after impact test, crown-shaped deformation attracts attention*.

Figure 3.28. *The copper bullet completely penetreted but not overall mass.*

Group (C): 450 °C -16h. 46.7 % potassium nitrate Armor glass tested by EN1063 level 2 (BR2) for 5 shooting 8 g bullet with 385 for 10 mm striking distance resulted all shots partial penetrated as shown in table (3.6) and figure (3.29), (3.30) shown front and back armour side after test. Figure (3.31) shown the 9 mm embedded bullet, and 7.62 copper jacket splits but no overall loss of mass.

Table 3.6. *Level 2 impact test conditions of Armor group (C).*

No. of shots	Type of bullet (mm)	Ballistic standard and striking distance	Speed (m/s)	Mass (g)	resu lt
1	9 x 19	EN1063-BR2	392	8±1	PP
2	9 x 19	EN1063-BR2	395	8±1	PP
3	9 x 19	EN1063-BR2	388	8±1	PP
4	9 x 19	EN1063-BR2	376	8±1	PP
5	9 x 19	EN1063-BR2	384	8±1	PP

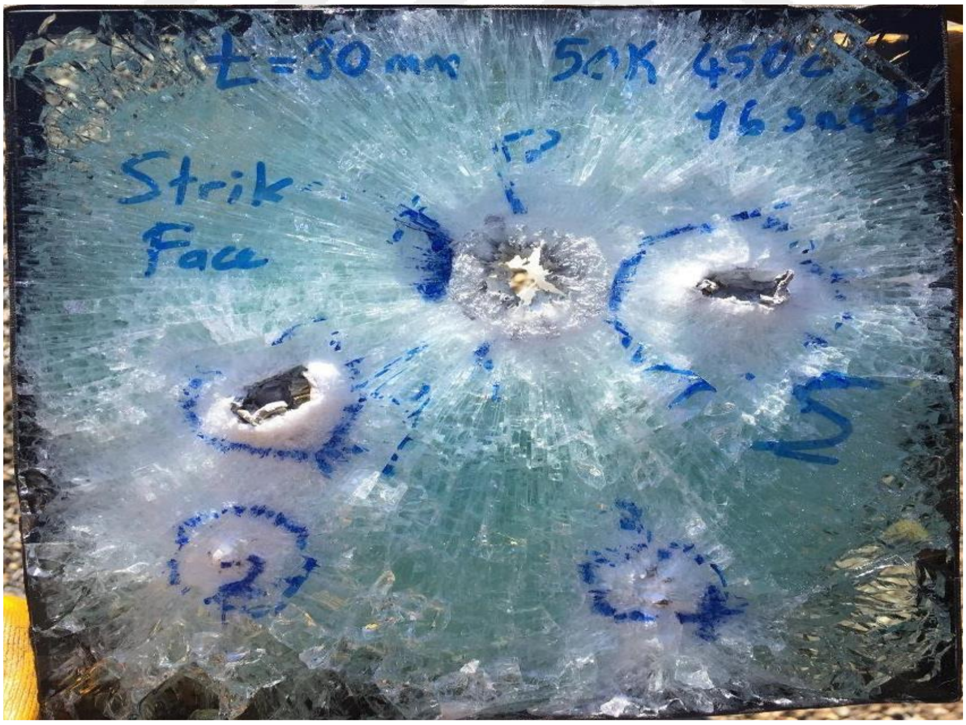

Figure 3.29. *Group (C) front side of armour glass after level 2 testing.*

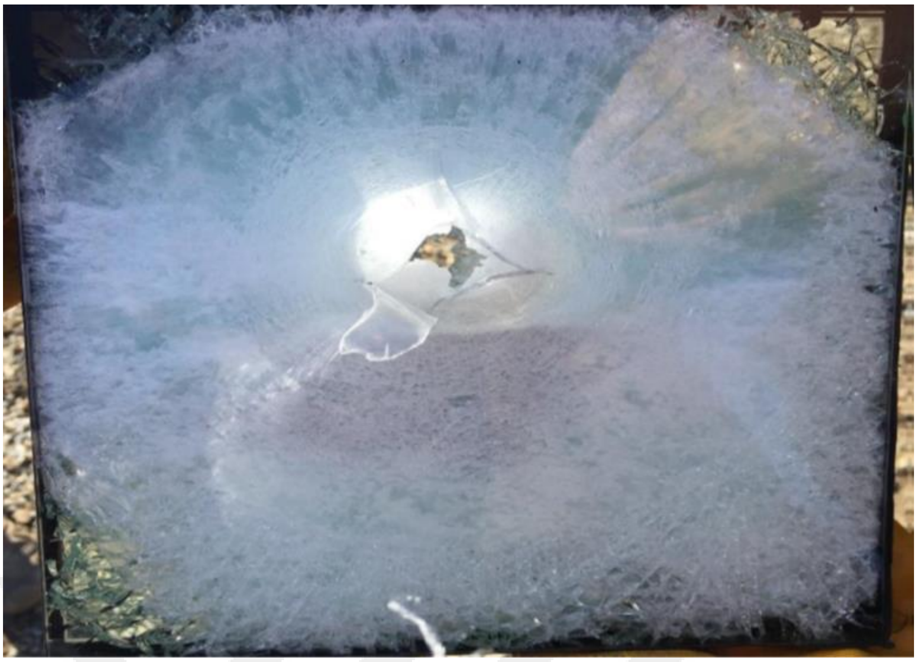

Figure 3.30. *Group (C) back side of armour glass after level 2 testing.*

Figure 3.31. *The embedded bullet and copper jacket splits but no overall loss of mass.*

Group (D): 450 ^{o}C-16 h.-63.7% potassium nitrate coated resulted partial penetrated for 8 shooting by level 2 for same conditions of test table (3.7) included results value, and figure (3.32) and (3.33) showing the front side and back side after impact test.

Table 3.7. *Level 2 impact test conditions of Armor group (D).*

No. of shots	Type of bullet (mm)	Ballistic standard and striking distance	Speed (m/s)	Mass (g)	result
1	9 x19	EN1063-BR2	392	8±1	PP
2	9 x19	EN1063-BR2	395	8±1	PP
3	9 x19	EN1063-BR2	388	8±1	PP
4	9 x19	EN1063-BR2	376	8±1	PP
5	9 x19	EN1063-BR2	384	8±1	PP
6	9 x19	EN1063-BR2	384	8±1	PP
7 (SWF) corner shot	9 x19	EN1063-BR2	368	8±1	PP
8 (SWF) corner shot	9 x19	EN1063-BR2	379	8±1	PP

Multiple shot resistance was observed as the best sample. The breaking pattern was typical and the same as the previous ones. Low trauma (Small amount of bulging) Shooting ranges between 10-25 mm.

Figure 3.32. *Group (D) armour glass front side after impact test.*

Figure 3.33. *Group (D) tempered armour glass back side after impact test.*

The test level 2 (BR2) showed partial penetrated bullet for all groups except group (A) showed bulging (CP). The bulge was due to close successive bullets. The group (D) showed higher performance in spite the bullets were in the corners.

The test level 6 (BR6) applied for only group (C), and showed (CP) completely penetrated bullet. Shim G.I. mentioned successful Armor glass in level 6 [69].

Contact time Increment recorded improve strength of Armor**.** Also increment in salt concentration recorded increasing in strength of Armor which explained above.

4. CONCULUSION:

The study was successful in finding a novel technique in ion exchange for glass strengthening. This study showed increasing of mechanical properties closed to other studies used traditional method of ion exchange. And improvement in the optical properties.

- Using this method showed better mechanical properties for thin glass compared to thick glass, although the limitation of thin glass tempering in high temperature.
- The increment of salt concentration showed minimal improvement in mechanical properties.
- Increasing the contact time up to 16 hours showed improvement of mechanical properties with cyclical pattern at 450 ^{o}C thick glass. Except in case of 500 ^{o}C where the best result was in 8 hours.
- The best tempering temperature in this study was at 500 ^{o}C where the glass strength after tempering increased up to 4.75 folds compared with untreated glass. The increment of tempering temperature up to 550 ^{o}C was disadvantageous in term of mechanical properties and optical properties.
- The laminated tempered glass with this method was suitable as bulletproof material. According to EN1063-BR2 standard but failed in EN1063-BR6 standard.

4.1. Recommendations:

- The study recommends to use 500 ^{o}C as a tempering temperature with 8 hours contact time, in regards to salt it's better to use powdered less than 500 μm to improve workability of paste.

The researcher of the study recommends other searchers to use microwave instead of electrical furnace and using lower contact time hoping to make this method applicable as a continuous method, like in case of thermally tempering.

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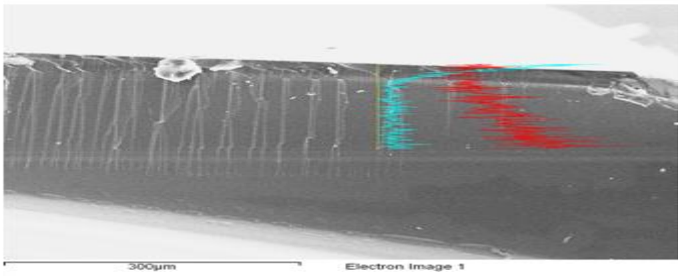
APPENDIX

Hardness Reading

Hardness of thin glass									
	lowest								highest
8hrs./30	471	471	526	526	590	591	607	608	608	633
8hrs./ 60	503	534	538	551	557	566	589	605	607	618
12hrs./30	477	541	561	567	598	603	615	619	641	668
12hrs./60	546	561	589	598	607	633	640	651	661	678
16hrs./30	497	546	566	581	601	613	641	663	689	698
16hrs./60	540	583	588	591	607	640	641	648	650	669
standard	401	487	513	523	529	567	569	578	583	603

Hardness of thick glass (4mm)										
	Lowest									Highest
8hrs./30	466	484	488	509	549	561	566	568	581	583
8hrs./ 60	494	523	526	551	553	560	576	581	591	594
12hrs./30	554	554	561	566	568	568	607	633	650	661
12hrs./60	544	560	562	563	566	571	573	578	581	594
16hrs./30	501	523	530	531	545	557	568	573	588	599
16hrs./60	538	546	555	561	583	591	598	602	618	641
standard	441	468	543	545	561	566	568	568	576	591



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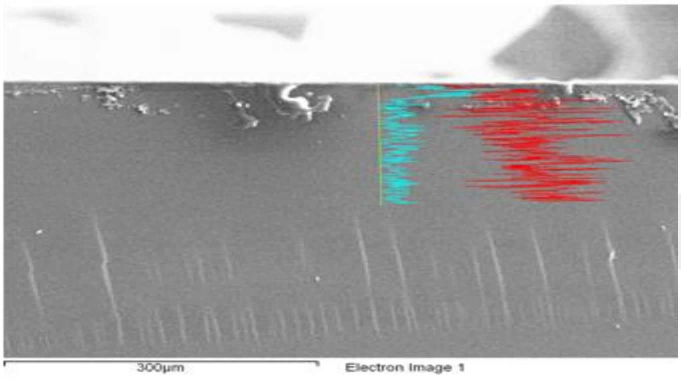

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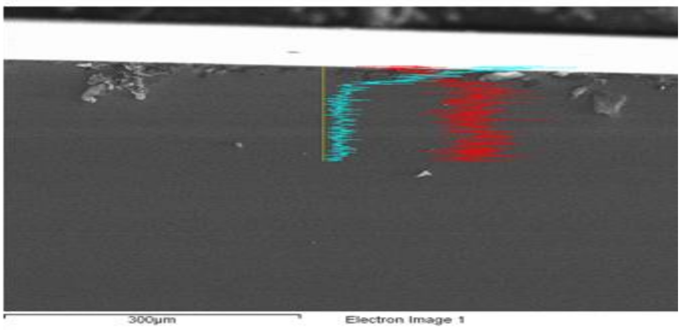

Line scan

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Summary:

Materials engineer specialized in ceramic materials precisely in glass. I have a good knowledge in CES program of materials selection and programs of design, and testing of materials. I can tolerate working under stresses and solving problems.Through education yaers I have good knowledg in heat treatment of metals and ceramics materials.

Skıll:

- Project management.
- Strong decision maker.
- Complex problem solver.
- Creative design.
- Innovative.
- Service-focused.

Experience:

- Work as construction Engineer2013-2015.Iraq/Baghdad.
- Work as QC Engineer for 6-month 2015. Iraq/Baghdad.
- Sales representative 2011-2013. Iraq/Baghdad.

Education:

Bachelor degree in: Materials Engineering – 2015 University of Technology/ Baghdad, UOT.
Turkish language: kırıkkale University 2017.
Master degree in: Materials Science and Engineering-2020 Eskisehir Technical University, ESTÜ.

Another Skill

Mechanical tests and some of NDT test, CES program of material selection, Sketch up, AutoCAD, origin lab data analysis and graphic, Microsoft.

Research:

1. Durability of Mortar Using Waste Glass Powder as Cement Replacement
2. New Generation Processing of Glass by Chemical Tempering.