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Hardening Glass to Resist Bullet Impact

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

اقْرَأْ بِاسْمِ رَبِّكَ الَّذِي خَلَقَ * خَلَقَ الْإِنْسَانَ مِنْ عَلَقٍ * اقْرَأْ وَ

رَبُّكَ الْأَكْرَبُ * الَّذِي عَلَّمَ بِالْقَلَمِ * عَلَّمَ الْإِنْسَانَ مَا لَمْ يَعْلَمْ *

طَبَقَ اللَّهُ الْخَطِيبِ

الخلاصة

في هذه الدراسة استخدمت ثلاث طرق للتقوية و هي المعالجة الحرارية (Thermal treating) و الإزالة السطحية (Etching) و عملية التصفيح (Lamination). إن الزجاج المستخدم هو زجاج الصودا- كلس المتوفر بكثرة. لقد تضمنت عملية المعالجة الحرارية تسخين الزجاج إلى درجات حرارة مختلفة (575°C , 600°C , 625°C , 650°C). في عملية التبريد تم الاعتماد على درجة حرارة الهواء الاعتيادي كدرجة حرارة هواء التبريد وكانت (17°C , 20°C , 35°C). لقد تمت عملية التبريد بثلاث طرق و هي أولاً بالحمل الحر (Free convection) و الثاني بنفث الهواء (Forced convection) و الثالث بنفث الهواء لفترة محددة ثم الإخماد بالزيت. إن أفضل الظروف للمعالجة الحرارية هي بتسخين الزجاج إلى درجة حرارة (650°C) و بعد أن يخرج من الفرن, يبرد بالهواء المنفوث بدرجة حرارة (17°C) لمدة ٢٥ ثانية و بعدها يغمر بالزيت.

لقد استخدم محلول مخفف من حامض الهيدروفلوريك (HF) لعمل حمام حامضي لمعالجة الزجاج سطحياً و بتراكيز مختلفة و هي (5% , 10% , 20% , 30% , 40%) HF. إن درجة حرارة المعالجة كانت (35°C). لقد كان أفضل أداء للزجاج المعالج بتراكيز محلول (5% , 10%) HF لمدة ٧٥ دقيقة, فبالإضافة إلى رفع المقاومة فإن الزجاج في هذه التراكيز يتمتع بشفافية جيدة مقارنة بالتراكيز الأخرى. إن عملية التصفيح تمت بكبس طبقتين أو أكثر من الزجاج مع طبقات من الاكريليك لجسم واحد باستخدام الايبوكسي كمادة لاصقة.

إن الاختبارات التي أجريت في هذه الدراسة هي اختبار الانحناء و اختبار الصلادة و اختبار المتانة بنوعيهما (الساكنة و الديناميكية) وأخيراً اختبار الصدم ببندقية كلاشنكوف.

لقد ظهر في اختبار الانحناء للمعالجة الحرارية إن مقاومة الكسر لأفضل طريقة هي 309.4MPa . أما للإزالة السطحية فكانت (136.2MPa , 162.2MPa) للتراكيز (5% , 10%) HF على التوالي. أما لعملية الجمع بين المعالجتين فكانت النتائج (276MPa , 315.7MPa) للتراكيز (5% , 10%) HF على التوالي.

إن صلادة الزجاج غير المعالج هي (549Kgf/mm^2), و بفعل المعالجة الحرارية أصبحت هذه القيمة (516Kgf/mm^2). أما الإزالة السطحية فقد رفعت هذه القيمة إلى (572.3Kgf/mm^2 , 578Kgf/mm^2) للتراكيز HF (10%, 5%) على التوالي. إن المزج بين المعالجتين قد رفع قيمة الصلادة إلى (552Kgf/mm^2 , 560Kgf/mm^2) للتراكيز HF (10%, 5%) على التوالي.

إن نتائج المتانة الساكنة بينت زيادة بالطاقة الممتصة كلما زاد عدد الطبقات في النموذج المصفح الواحد و أيضا عند استخدام الزجاج المعالج بدلا من الزجاج الغير معالج. لقد أظهرت نتائج اختبار المتانة الديناميكية نفس تصرف اختبار المتانة الساكنة ما عدا إن الأخيرة أعلى من الأولى.

إن اختبار الصدم اظهر إن النموذج المكون من خمس طبقات زجاج معالج (بإزالة سطحية وبصورة حرارية) و خمس طبقات من الاكريليك بحيث إن السمك النهائي يكون 40mm , قادر على أن يصد طلقة الاكلاشنكوف من مسافة 10m و بكثافة مساحية مقدارها 78.7Kg/m^2 .

ABSTRACT

In this study three techniques were used to strengthen glass which were heat treatment, etching, and lamination process with a polymer. Soda-lime glass is used as a raw material for this purpose. In heat treatment process, the glass was heated to different temperatures which were (575°C , 600°C , 625°C , and 650°C). The temperature of the cooling air was the same of the ambient. Air cooling temperatures were (17°C , 20°C , and 30°C). Cooling process was conducted by three ways which were first by free convection, second by air jet (forced convection) and finally by air jet to limited time then quenched in oil. The best performance to strengthen the glass by heat treatment was heating the glass to 650°C , then taking it out of the furnace and cooling it by air jet of temperature of 17°C for 20 seconds, then quenching it in oil.

Diluted Hydrofluoric acid (HF) bath was used to etch this glass with concentrations of (0%, 1%, 2%, 3%, and 4%). The temperature of etching bath was 30°C . The well performance for etching was at (0% and 1%) for 70 minutes where the glass transparency of these concentrations was the best among the others.

Lamination process was done by compressing glass plies with acrylic sheets to form a body using epoxy as a glue material. Mechanical properties such as bending, hardness, dynamic toughness and finally shooting by a Kalashnikov rifle were done in the study.

For heat treatment, three point bending test shows the best value was 309.4 MPa as a fracture strength. For the etching the fracture strength of glass was (162.2 MPa and 136.4 MPa) at (0% and 10%) HF respectively. The combination of heat treatment and etching gave fracture strength of (276 MPa and 310.7 MPa) at (0% and 10%) HF respectively.

The hardness of annealed glass was 049 Kgf/mm². Heat treatment of the glass was decreasing the glass hardness to 016.7 Kgf/mm² at best conditions of this process. Etching process was raising slightly the hardness of the glass (072.3 Kgf/mm² and 078 Kgf/mm²) at (0% and 10%) HF respectively. The combination of heat treatment and etching was giving enhancement for hardness where it became (002 Kgf/mm² and 060.3 Kgf/mm²) at (0% and 10%) HF respectively.

The results of static toughness of laminated glass showed an increment in absorbed energy as the number of glass and acrylic layers was increased. The static toughness of treated (heat treatment with etching) glass with acrylic was higher than that of the untreated one. Dynamic toughness showed an increment in absorbed energy by the laminated glass as the thickness of laminated glass was increasing. The absorbed energy in dynamic toughness was much higher than that of the static toughness.

Shooting test showed that the sample of laminated glass consisted of five layers of treated (heat treatment with etching) glass with five layers of acrylic can defeat the Kalashnikov's bullet of caliber of 7.62 mm from a distance of 10 m.

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CHAPTER ONE

Introduction

A glass is defined by American Standard for Testing and Materials (ASTM) as "an inorganic product of fusion which has been cooled to a rigid condition without crystallization" [١,٢]. It is an inorganic substance with the same structure of liquid state of that substance. It has a short-range order instead of a long-range order as in crystalline materials [٢,٣,٤,٥].

The characteristics that make glass desirable and useful are its hardness and transparency and other properties [٦]. Silica is the most important constituent of glass, but other oxides are added to reduce the melting point in order to simplify processing or to change the physical characteristic of glass. Because of the unique properties of glass, it can be cast, rolled, drawn, and pressed like a metal, in addition it can be blown [٧].

The theoretical tensile strength of glass is around (١٠٠٠٠ MPa) while the measured tensile strength of glass is much lower around (٣٣ MPa). This large discrepancy is due to small flaws in the glass, particularly those on the surface which are subjected to a tensile stress. To remove the flaws from the surface

of a piece of glass, there are two ways; either by polishing or by chemically etching [1,9].

The compressive strength of glass is very high but the tensile strength is low compared to most metals. Tensile and compressive stresses can be controlled by both mechanical and chemical methods (Heat treatment and ion-exchange).

In both methods compressive stress is created in the surface layers [1,9]. There are essentially four types of glass categorized by the amount of heating and cooling rates used in the manufacturing process [10]:

- a. Annealed float glass.
- b. Heat-treated glass.
- c. Tempered glass.
- d. Chemically strengthened glass.

In general, a higher temperature coupled with a more rapid cooling rate in the production will produce glass that is stronger and more resistant to breakdown [10].

Modern architectural designs required glazing materials that provides enhanced levels of security and safety performance properties. These properties include resistance to ballistics, blasts, hurricanes/cyclic wind pressure and physical attacks. Applications may also required desirable properties such as sound reduction, solar and thermal control [10].

One of the solutions for these requirements is a laminated glass which is a multiple plies of glass interlayered by resins and/or plastic glazing materials (such as polycarbonate sheet or acrylic) which are often complex in nature.

They are designed to provide specific levels of performance. In all their application, the thickness and configurations of the components play an important role. The thickness of the glass portion of the laminated glass appears to be a more apparent degree of green. It is ranging from the simple glass- Polyvinyl Butyral (PVB) laminates in windows to advanced glass polycarbonate laminates in bulletproof glass [11, 12]. It can be manufactured from heat-strengthened and fully tempered or chemically tempered glass for additional benefits, and this will follow cost [13, 14].

Almost all security glasses are laminated glass which can be designed to resist attacks by a wide range of weapons. They have two main requirements which are [15]:

1. To resist penetration by a special bullet.
2. To avoid spalling or flying shards of glass leaving the rear face, as a result of the impact.

Bullet-resistant laminated glass can typically meet both of these requirements [15]. Table (1-1) illustrates the type and caliber of weapon, bullet weight, bullet velocity, thickness, and areal density for glass-clad polycarbonate [16]:

Table (1-1) General properties of many targets [16]

Weapon	Caliber	Weight, g	Velocity, m/s	Thickness, mm	Areal density, Kg/mm ²
Handgun	9mm	8	410	16	33
Handgun	9mm	7.5	392-410	18	37
Handgun	0.44 in.	10.6	410	18	37

Handgun	0.307 in.	10.2	43.	19.0	38
Rifle	7.62	7.77	710-710	33.0	72.0
Rifle	7.62	9.0	830-840	36	78
Rifle	7.62	9.70	820	72.0	172.8

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction:

Several papers have been published in the literature concerning the strengthening of glass. They include etching, heat treatment, chemical treatment and laminated glass. These papers will be viewed to show the evaluation of glass technology of the related matter:

1. Literature review concerns with Etching

Ernsberger (1960) has shown that the tensile strength of glass increases when glass is etched in (5%) hydrofluoric acid (HF) solution, and shown decreasing in the density of crack patterns when the time of etching is increased [17].

David and Alfred (1986) have studied the weakening effect of insoluble reaction products that make contact with but are not deposited on the glass surface. They have used rods of soda-lime glass and etched it to a constant depth ($20 \pm 1 \mu\text{m}$) in acid bath of (1 wt% HF, 1 wt% H_2SO_4 , the rest water). They have used mixer and have shown that the impact of the insoluble reaction products on the etched surfaces can cause strength degradation [18].

7. Literature review concerns with ion-exchange

Shibin et al. (1995) have investigated the thermal shock of phosphate laser glass. They have concluded that there is an increasing in the thermal shock property by a factor of (2) at the glass treated by the ion exchange in a molten of KNO_3 and NaNO_3 . This result reflects a dramatic enhancement of the thermal loading capacity for the glass without affecting the optical properties of this glass [19].

Peitl and Zanotto (1999) have investigated the thermal shock of borosilicate glass which is subjected to ion exchange in molten of KNO_3 . The samples of this glass are held in the molten bath at 446°C and 480°C for periods of 2, 4, 8, 16, 32, and 64 hours. They have concluded that the strength of thermal shock of this glass increased where the number of cracks are decreased in the treated sample [20].

Hoikwanlee et al. (2003) have studied the flexural strength of chemically hardened glass by using single ion exchange. They have used samples of soda-lime glass in a molten of KNO_3 at temperatures ranged from 470°C to 560°C for periods varied (0.5-4) hours then held at 400°C for 24 hour. The strength of glass has measured by three point bending. The strength of untreated glass was 88.3MPa . They have found that the maximum value of strength was 617.8MPa after an ion-exchange process at 470°C for 1 hour then, at 400°C for 24 hour. After ion exchange treatment the strength increased by factor of 6 [21].

A. K. Varshneya and Glenn A. Dumais (1980) have studied the ion exchange process in a bent samples of soda-lime glass. KNO_3 has used as a bath of treatment with stirrer. The interval of processed glass is 4 hours at

400°C, 450°C and 500°C. They have showed that the maximum diffusion occurred from the tension side while the minimum diffusion of ions occurred from the compressive side, and the neutral side is between them. These observations support the notion that extremely applied stresses can alter the average site size for cation diffusion in glass [22].

3. Literature review concerns with lamination of glass

Veer et al. (1997) have investigated the possibility of mechanically or chemically cohesion of glass/glass, glass/metal and glass/polymer. Several techniques of joining are used which are: mechanically by bolts, chemically by adhesives, and by soldering, and physical joining by welding. They have concluded that the best joining technique of glass laminate can be done chemically by adhesives [23].

Veer and de Vries (2001) have studied the behavior of transparent laminated composites which are prepared with different configurations ranging from one layer of glass to layers of segmented glass with polycarbonate foils and they simulated numerically with finite elements method by using ANSYS program. They have found that the transparent laminated composites have much better mechanical properties comparable to aluminum and they have high impact resistance [24].

Hobbelman et al (2001) have studied experimentally and analytically the behavior of combination glass layers with polycarbonate layers. They prepared many samples with dimensions, height 38mm, length 420mm. The thickness and the number of polycarbonate layers have varied. 3D FEM program ANSYS has used to analyze the behavior of these composites. They have found an agreement between the experimental and analytical work [25].

ξ. Literature review concerns with glass against bullets

Hazell and Armstrong (2001) have done experimental and numerical programs to investigate the mechanics of perforation of the 7.62 x 39 mm NATO ball round through multiple glass systems. Each system consisted of four float soda-lime glass plates with either 1.0, 1.5, or 2 mm spacing between each plate. The thickness of glass plate varied from 3 mm to 12 mm. They have proved that the simulation does not predict the full dynamic response of the materials. Experimentally they have showed that the spacing between each plate results in little difference to the residual momentum of the penetrating core. The system of four 12 mm layers of glass with 1.0 space has stopped the projectile with depth of penetration 40 mm [26].

Richards et al. (1999) have studied the performance of float soda-lime glass/polycarbonate laminates experimentally and numerically by using AUTODYN program. They have used three thicknesses of this glass which are 3 mm, 6 mm, and 12 mm. The final thickness of the system (composite) is 20 mm. A 7.62 x 39 mm NATO ball round is used as a standard projectile. They have found that the system of 12 mm thick glass with 6 mm polycarbonate has stopped the projectile. The results of the numerical work is relatively the same of the experimental work [27].

φ. Literature review concerns with glass hardness

Everstiejin et al. (1960) have reported that annealed glasses exhibit higher hardness values in comparison with quenched glass. Because the quenched glass have a more open structure (low density) than that annealed glass [28].

Yoshioka and Yoshioka (1995) have constructed an apparatus for Vickers indentation of soda-lime glass with indenter velocity of (0.2-1 m/s) and load of

2.0N to 13.3N. A camera has monitored the size of the growing impression during the loading cycle. They have concluded that the fastest growth in an indentation size occurred at initial loading and the initial penetration rate was typically 0.2 times the collision velocity [29].

It is clear from the preceding papers that there were four techniques to strengthen glass which were:

1. Etching the glass surface.
2. Strengthening by chemical ion exchange.
3. Strengthening by tempering.
4. Strengthening by laminating.

2.2 The Aim of The Study:

The present study will be concerned with strengthening soda-lime glass to resist bullet impact. To do this goal, most of methods available in literature have to be evaluated especially the methods which are not counteract each other. The strengthening of glass by tempering, etching, mixed them together and laminating were adopted.

CHAPTER THREE

THEORETICAL PART

3.1 Introduction:

Glass becomes a popular commonly in the growth of civilization. When a liquid is cooling rapidly enough such that the atoms do not have enough time to rearrange themselves in a crystalline pattern before their motion is arrested then glass is formed. The density and mechanical properties of glass are solid-like. Unlike a crystal, glass may not be represented by a simple chemical formula. In the absence of any external effect such as mechanical, thermal and magnetic the properties of glass are isotropic like those of a typical liquid [14,30].

3.2 Glass structure

Silica glasses and minerals are not composed of discrete molecules but they are three dimensional connected networks. The basic structure unit of the silicate network is the silicon-oxygen tetrahedron in which a silicon atom is bonded to four larger oxygen atoms. Silica is belonged to tectosilicate class in which all the oxygen ions in each (SiO_4) tetrahedron is shared with neighboring tetrahedra. This result in a stable and strongly bonded structure in which the ratio of Si : O is 1 : 2 as shown in Figure (3-1 and 3-2) [14,30].

The units of silica tetrahedron are linked at the corners by sharing of one oxygen atom between two silicon atoms. All four oxygen atoms from one tetrahedron can be shared with four other tetrahedra to give a three-dimensional network. These shared oxygen atoms are called "bridging

oxygen". In pure silica glass or pure silica minerals, such as quartz, the ratio of silicon to oxygen is 1:2 and all oxygen atoms are bridging. Some atoms when presented in glass, such as sodium, are ionically bonded to oxygen. This disrupts the continuity of the network, since some of the oxygen atoms are no longer shared between two tetrahedra, but they are bonded to only one silicon atom. This type of oxygen atom is called a "non-bridging oxygen" [14].

There are three major constituent groups of oxides that may be found in glass batch [15]:

1. The forming oxides, which are forming glasses by themselves joining together as previously mentioned silica glass structure as illustrated in Figure

(3-2). These oxides are B_2O_3 , P_2O_5 etc....

2. Modifiers or alkali oxides of low-valence elements such as sodium and potassium. They tend to break up the continuity of the chains, but they can be added in limited amounts. Addition of such oxides leads to a lower melting temperature and simpler processing as shown in Figure (3-3). Examples of such network modifiers are Li_2O , Na_2O , and K_2O .

3. The intermediate oxides which do not form glasses by themselves but join the silica chain to maintain a glass as illustrated in Figure (3-4). Examples of such intermediate oxides are PbO and Al_2O_3 .

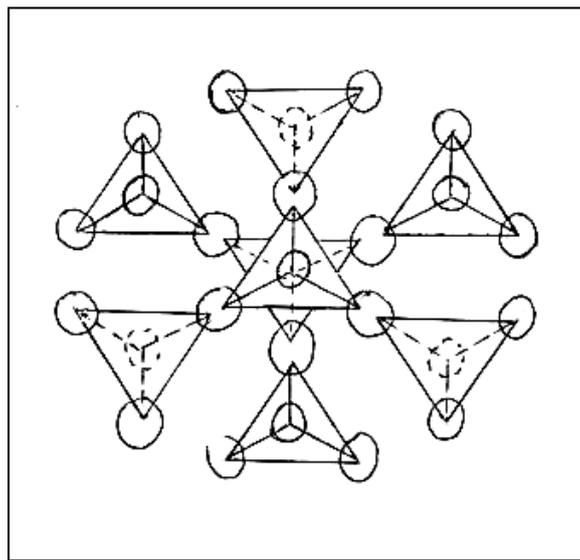


Fig.(3-1) Two-dimensional sketch of a silica network in crystalline material [3-1].

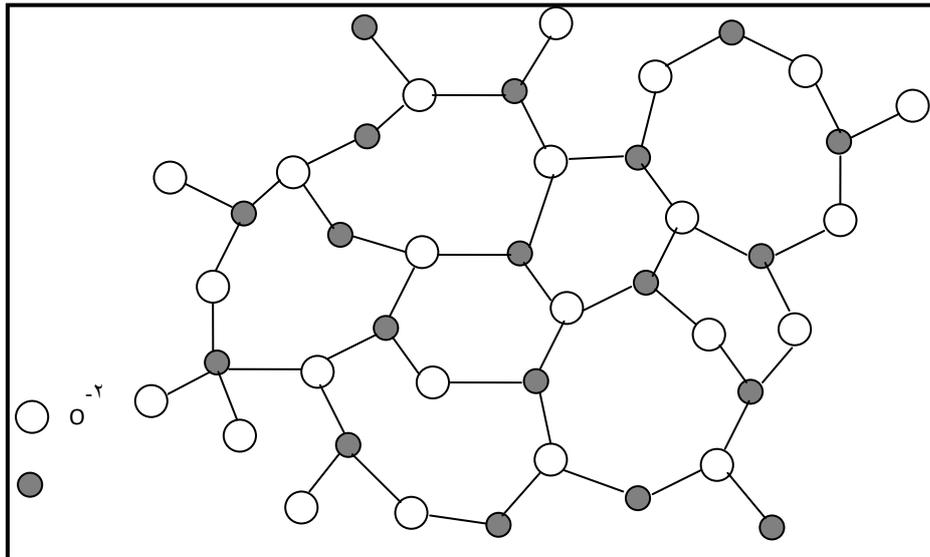


Fig.(3-2) Two-dimensional sketch of silica glass form [3].

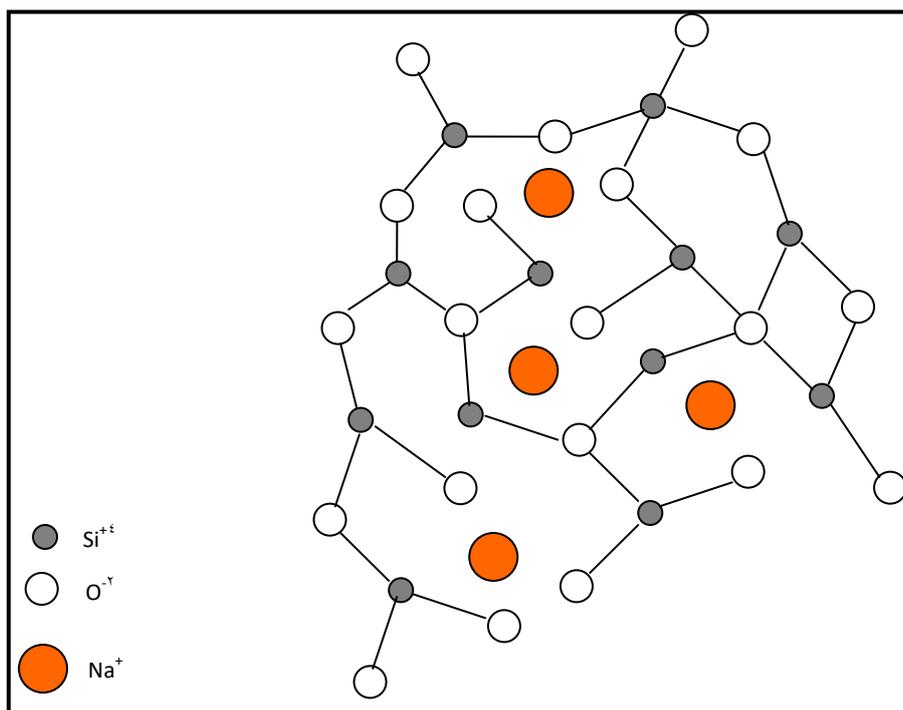


Fig.(۳-۳) Two dimensional schematic of the silicate glass structure in the presence of modifier ions as (Na) and the formation of non-bridging oxygen [۴].

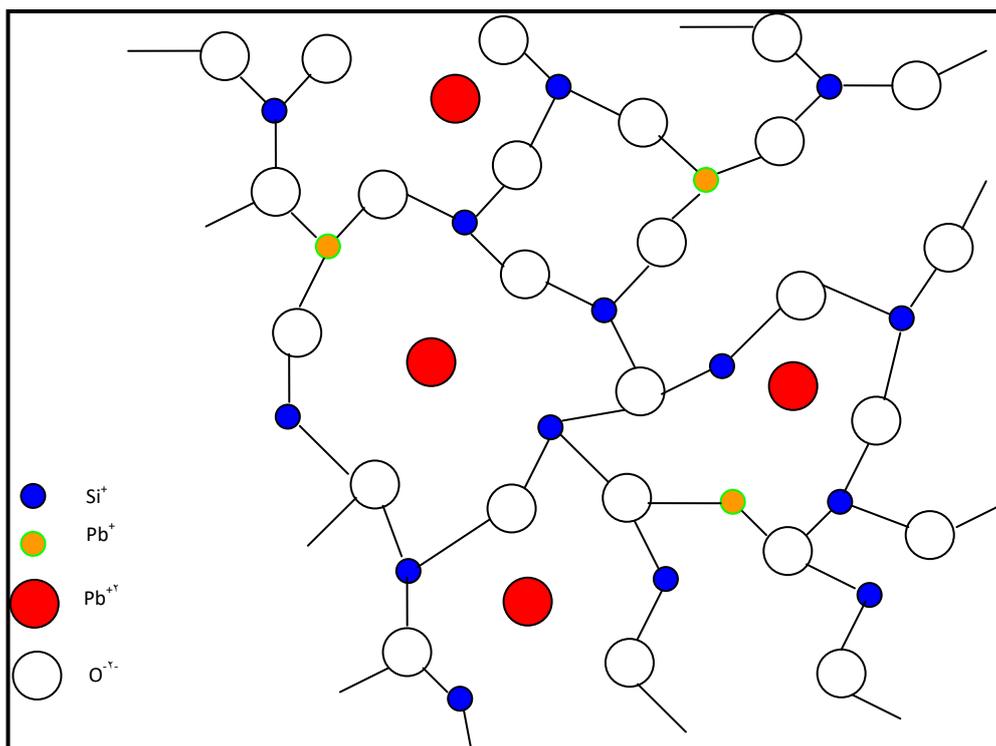


Fig.(3-4) Schematic of intermediate lead oxide occurs both in the silica network as (Pb⁴⁺) and as a modifier in internal position (Pb²⁺) [4].

3.3 Types of Glasses

3.3.1 Soda-Lime Glasses(SLG)

Most commercial soda-lime glass compositions are based on a eutectic composition in the (Na₂O-CaO-SiO₂) phase diagram located at about 22wt%Na₂O, 9wt%CaO, and 69wt%SiO₂. A typical composition of soda-lime glass is (10Na₂O, 10CaO, 2Al₂O₃, 68SiO₂ wt%) [13]. Soda-lime glass is almost insoluble in water. Its density is about (2.5g/cm³), hardness is about (6to7) on the Mohs scale, stiffness(E) is about 70GPa and design strength above 50MPa. It is transparent and electrical insulator with a good dielectric strength. It has a high thermal expansion (about 9.0x10⁻⁶/°C) [6,32]. It is used to make sheets for windows, bottles, jars and household lamps. After some treatments, this glass is used to manufacture safety glass, windshields and antiballistic sheets [30].

Tables 3-1 and 3-2 explain fracture toughness and tensile strength of some material in comparison with soda-lime glass [33].

Table (3-1) [33]

<u>Material</u>	<u>Fracture toughness</u> K _{IC} (MPa m ^{-1/2})
Pure ductile metals	100 – 300
High strength steel	50 – 100
Mild steel	100

Titanium alloys	55 – 110
Aluminum alloys	23 – 40
Cast iron	6 – 20
GFRPs	20 – 60
Boron fiber epoxy	46
Polypropylene	3
ABS polystyrene	4
Nylon	3
Acrylic	0.9 – 1.4
Epoxy	0.5
Silicon Nitride, Si ₃ N ₄	4 – 5
Silicon Carbide, SiC	3
Alumina, Al ₂ O ₃	3 – 5
Soda-lime glass	0.7 – 0.8

Table (3-2) [33]

<u>Material</u>	<u>Tensile strength (MPa)</u>
Low alloy steel	680 – 2400
Nickel alloys	400 – 2000
Copper	400
Aluminum alloys	300 – 700
CFRPs	100 – 300

Boron fiber epoxy	20 – 173.
Polypropylene	33 – 36
Acrylic	11.
Epoxy	30. – 120.
Nylon	100.
Diamond	–
Silicon Nitride, Si ₃ N ₄	–
Silicon Carbide, SiC	–
Silica glass, SiO ₂	–
Alumina, Al ₂ O ₃	–
Mullite	–
Soda-lime glass	–

3.3.2 Other glasses

A wide range of glass quantities are manufactured for many purposes. Some of them are listed below:

3.3.2.1 Lead Silicate Glass

When the lime (CaO) is substituted by lead oxide (PbO) in the soda-lime-glass mixture, the result is a glass with a higher refractive index. This glass is used for vases and other reflected uses. It absorbs gamma and x-rays, so it is used for shielding those rays in technical equipments [6, 14, 30].

3.3.2.2 Borosilicate Glass

This glass has a composition of (81 SiO₂. 13 B₂O₃. 4 Na₂O. 2 Al₂O₃ wt%). It has a low coefficient of thermal expansion (about 1.7x10⁻⁶/°C), high melting

temperature (transformation rang), and excellent chemical durability. Therefore it is used for laboratories and chemical ware, but also for manufacturing of cooking ware like Pyrex and Kimax [३,३०].

३.३.२.३ Aluminosilicate Glass

A typical composition of this glass is (68 SiO_2 , $20 \text{ Al}_2\text{O}_3$, 16 CaO , $0 \text{ B}_2\text{O}_3$, $1 \text{ Na}_2\text{O}$ wt%). This glass has a good electric properties, good resistance to chemical attack, and good use for high temperature applications. It is used for electrical and electronic applications [३०].

३.३.२.४ Silica Glass

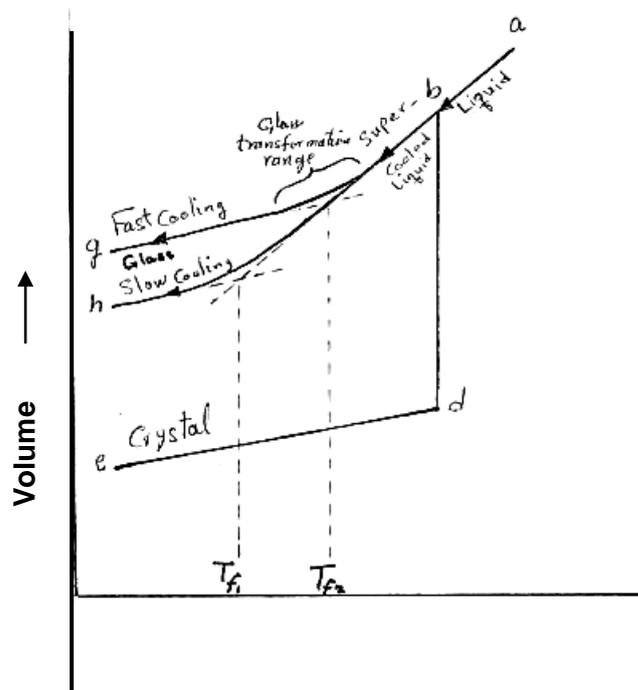
Silica glass is the glassy form of the chemical compound SiO_2 . It has the highest refractoriness, lowest coefficient of thermal expansion among glasses ($0.05 \times 10^{-6} / ^\circ\text{C}$), very high chemical resistance, high optical transparency, and low dielectric constant. Major applications of this glass group are lighting, semiconductor industry, optical use, high-energy laser optics, and spacecraft windows [३०].

३.४ Glass Transformation

The glassy state differs from crystalline materials where the change of crystalline materials during cooling is converted from disorder to order structure, but in the non-crystalline materials the change from disorder to disorder structure with less empty spacing [४].

३.४.१ The Effect of Cooling Rate

The behavior of glass under cooling can be explained by the relationship between volume and temperature as shown in Figure (3-9). At glass melt start to cool from high temperature (a), the volume shrinks along the line (ab). When there is a probability of crystalline, the liquid will solidify by creating nucleus and growth these nucleus to crystals without decreasing in its temperature (line bd) then mass shrinks along the crystal state line (de) on further cooling. In the absence of crystalline, the liquid at (b) passes into a super-cooled liquid state without the appearance of crystals. Further cooling, it becomes more and more viscous in a continuous manner with decreasing temperature and the path will be either (bg) or (bh) depending on the cooling rate. There is no definite temperature at which the glass liquid transforms to a solid as happened with crystalline materials. A slight decrease in slope of curve occurs at what is called "glass transition temperature" (T_g), or "fictive temperature" (T_f). Below this temperature, the material is considered to be a glass; above it (T_g) will be a super-cooled liquid, and finally a liquid [30, 34].



Temperature, °C →

Fig.(3-9) Specific volume versus temperature behavior of crystalline and non-crystalline materials [30].

3.4.2 The Effect of Temperature on Glass Viscosity

The functional dependence of viscosity on temperature has been measured in a number of glass-forming liquids. It is generally accepted that the relationship between temperature (T) and viscosity (η) in a glass is reasonably represented by Vogel-Fulcher-Tammann (VFT) empirically derived equation, dating back to the 1920's [4,30]:

$$\log_{10} \eta = -A + B/(T-T_0) \quad (3-1)$$

Where: η = the viscosity in Pa.s (N.s.m⁻²) or in poise.

A, B and T_0 = constants

T = the temperature of interest (°C)

Where base 10 logarithms are used because the large numbers are involved. The three constants A, B and T_0 in above equation can be determined for a

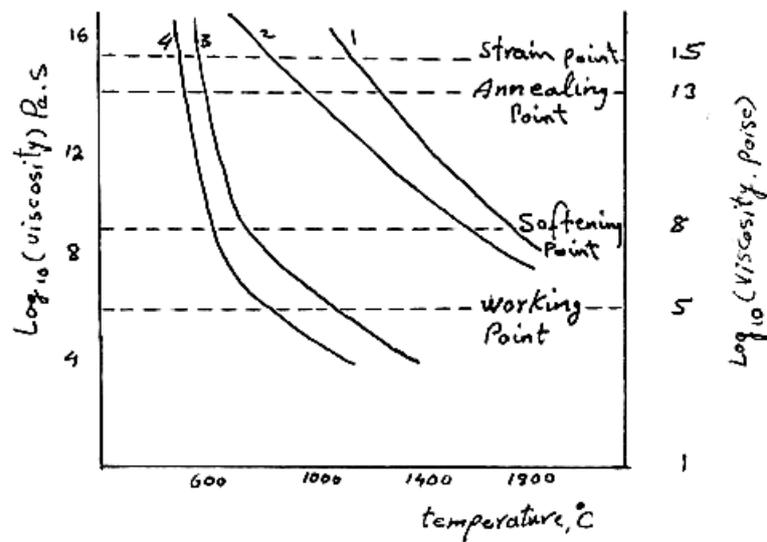
particular glass from three known temperature-viscosity points spanning at temperature rang. Glass manufacturers often tabulate the values of A, B and T_0 for their products. The various steps of glass forming are determined by using the viscosity temperature (ηT) curves. Five viscosity reference temperatures are used as below [4, 30, 36]:

1. **The melting point;** corresponds to the temperature at which the viscosity is 10^8 pa.s (10^8 p); the glass is fluid enough to be considered as liquid.
2. **The working point;** represents the temperature at which the viscosity is 10^7 pa.s (10^7 p), in the rang of this temperature the glass is readily drawn or pressed.
3. **The softening point;** the temperature at which the viscosity is 10^6 pa.s (10^6 p), is the maximum temperature at which a glass piece may be handled without causing significant dimensional alteration.
4. **The annealing point;** is the temperature at which the viscosity is 10^7 pa.s (10^7 p). At this temperature, atomic diffusion is sufficiently rapid that any residual stresses may be removed within about 10 min..
5. **The strain point;** corresponds to the temperature at which the viscosity becomes 10^8 pa.s (10^8 p); for temperatures below the strain point, fracture will occur before the onset of plastic deformation. The glass transition temperature will be above the strain point.

The temperature at which each these points occur depends on glass composition [4].

The relationship between viscosity of glass and its temperature is very important from the manufacturing point of view because it determines the

melting conditions, the time and temperature required to homogenize a melt, the working and annealing temperatures, the rate of devitrification and the critical cooling rate, as well as the temperature of annealing of residual stresses. Figure (۳-۶) shows this relationship [۴].



۱. silica glass

۲. ۹۶% silica glass

Fig.(۳-۶) Relationship between temperature and viscosity for many types of glass [۳۲].

۳.۵ Safety Glass

Safety glass can be defined as a glass which has passed specific impact tests and must either not break or break in a manner that will not cause injury. There are three types of safety glass which are toughened glass, laminated glass, and wired glass. All these glasses will be discussed as follows:

३.०.१ Toughened glass (Fully tempered glass)

Toughened glass is obtained when a sheet of glass is heated to approximately १००°C in a furnace then chilled rapidly by cold air blower on both surfaces. Toughened glass is four to five times stronger than float glass and, if it is broken, it disintegrates into small fragments with dulled edges, which are unlikely to cause serious injury [३१,३२].

३.०.२ Laminated Glass

Laminated glass consists of two or more thin sheets of glass separated by layers of a plastic or resin material. There are two main types of laminating glass which are Polyvinyl Butyral and resin that will be discussed below:

a. Polyvinyl Butyral

This is the more commonly used variety. Two or more sheets of glass are bonded together with one or more layers of Polyvinyl Butyral (PVB) (a plastic interlayer in sheet form). The principal benefit of laminated glass is their performance under impact. The glass may break but any broken fragments will firmly bond to the interlayer. The interlayer also absorbs impact energy, reducing the risk of penetrating the panel. The interlayer in laminated glass provides two additional benefits: sound transmittance is reduced, particularly at the higher frequencies and ultra-violet radiation (between ३२० and ३८० nanometers) is reduced about ९१%. [३१]. Laminating glass is १०% to १००% as strong as annealed glass of the same thickness depending on exposed temperatures, plate size, stiffness and load duration [३२].

b. Resin

Resin laminated glasses are manufactured by pouring liquid resin into a cavity between two sheets of glass which are held together until the resin cures. This method is ideal for laminating glass having a heavily textured or patterned surface. Not all resin laminates have safety performance. They are principally used for decorative and acoustic purposes, where safety performance is normally of secondary importance [37].

3.5.3 Wired Glass

It is annealed glass which contains a wire mesh embedded within the glass. It is intended to be used to safety building code requirements for fire protection. The impact resistance and breakage pattern of wired glass is similar to annealed glass. When the glass is broken, the embedded wire mesh helps to hold broken glass pieces together and keep them in the window frame. The visual quality of the glass is impaired by the wire mesh but this has traditionally been accepted in low-cost fire resistance and roof glazing [39].

3.6 Strengthening of Glass

Normal glass has a high value of compressive fracture strength, but it has moderately low value of tensile fracture strength due to occurring flaws on its surface. The strength of glass tends to be limited by tension because of propagation cracks that lead to fracture of the glass. To enhance the strength of bulk glass, the surface of glass should be compressively prestressed. Any applied load must then neutralize the compressive pre-load before potentially damaging tensile stresses in the surface. The usable strength in the glass is thus increased by the amount of the compressive stress [30, 40]. Two techniques are employed in strengthening of glass one physical and the other

is chemical. Both techniques are based on the fact that when the surface has the slightest imperfection (minute scratch), glass remains extraordinarily strong in compression but it becomes weak in tension. Thus, the strengthening treatment consists of prestressing a glass object by inducing compressive strains in its exterior, and thereby enabling it to counteract any tensile stresses which develop under tension [32]. The two techniques will be discussed below.

3.6.1 Physical Prestressing

The principle employed in the heat treating process is to create an initial condition of surface and edge compression. This condition is achieved by first heating the glass, then cooling the surface rapidly. This leaves the core of glass relatively hot compared to the surfaces. The cooling process for the core will make the surfaces and edges in compression state [41]. There are two types of the physical prestressing: heat strengthened glass and fully tempered glass.

3.6.1.1 Heat Strengthened Glass:

It is produced by heat treating annealed glass under regulated thermal conditions. In this process, annealed glass has been cut to size and it is heated in a furnace controlled between (293-840°C) and then cooled by air. Sudden cooling causes a compression envelope around the glass surface and edges and balanced tension stress within the glass itself. This equilibrium of stresses increases the strength of the glass to approximately two times than the original annealed glass [38]. Figure (3-7) illustrates the characteristic of stress profile produced by heat strengthening. Surface compression in heat strengthened glass ranges from 21 to 69 MPa. When it fractures, the fragments are larger than tempered glass but smaller than that an annealed glass fracture [38]. The fragments sometimes remain in the frame. It is suitable

for applications where annealed glass could be used but additional strength and thermal endurance are desirable [14]. It is not a safety glazing product and therefore should not be used where safety glazing is required. Glass thicknesses from 1/8 inch through 1/2 inch are commonly subjected to the process of heat strengthened [15].

3.6.1.2 Fully Tempered Glass

It is a heat treatment that can be applied to a certain type of glass, and it is accomplished by introducing surface compression into the glass [16]. In this process, the temperature of the glass is raised to a value in the neighborhood of the softening point. Then the glass is removed from the heating furnace and the surfaces are quickly chilled. Several chilling media can be used, including blast of air or bath of oil [17, 18].

The exterior layers of glass are cooled and contracted while the interior of the glass is relatively viscous. When the interior shrinks, the surfaces are rigid and they are compressed by the contraction of the interior. Figure (3-7) shows the characteristic stress profile produced by tempering. The exact shape of this curve depends upon the temperature range over which the rapid chilling takes place.

Fully tempered glass is considered a safety glazing material and it matches the requirements of American National Standard Institute for Safety Glazing Materials Used in Buildings (ANSI Z97.1). It has a higher impact resistance as order about four times than that of annealed glass [14]. When it does fail, the stored mechanical energy is instantly released causing rapid bifurcation of the advancing crack fronts. This yields a dicing form where the pieces do not have acute-angle corners. They are not expected to cause serious injury by sharp cut

edges [३०]. Because of its prestressed condition, tempered glass cannot be cut by any means except the width and length can be cut by grinding. For this reason, the glass must be cut to final size prior tempering. Full penetration of the compression layer will produce instantaneous and total fragmentation of tempered glass [१६].

The stress distribution through the thickness of tempered glass is approximately parabolic and the thickness of the surface compression layer is considerably greater than the depth of the most severe flaw [१६,३०].

The color, clarity, chemical composition and light transmission which are the characteristics of tempered glass remain essentially unaffected or unchanged after heat treating. Likewise, specific gravity, expansion coefficient, softening point, thermal conductivity, solar optical properties and stiffness remain unchanged by heat treating process. The only physical properties changed are improved flexural strength, tensile strength, improved resistance to thermal stress and thermal shock [६२,६३].

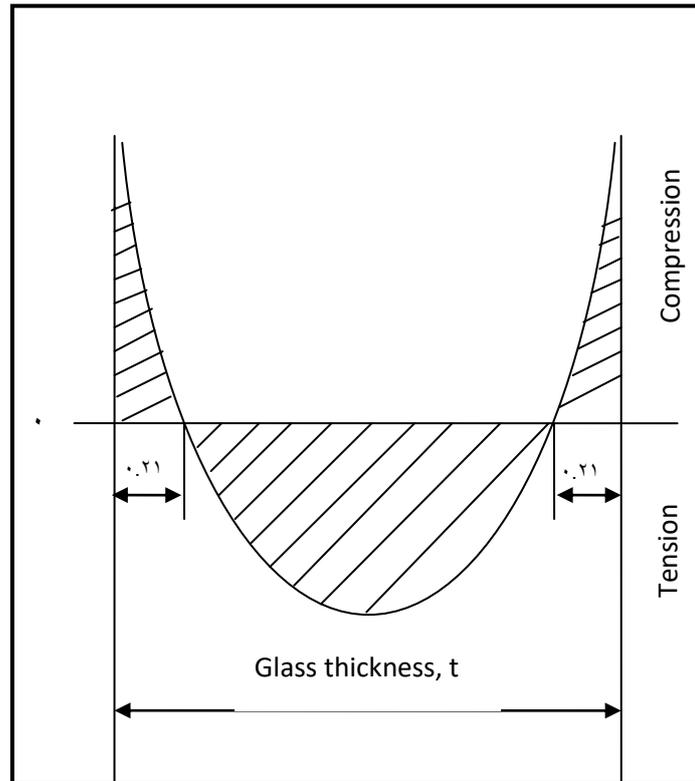


Fig.(۳-۷) Characteristic stress profile in heat strengthened and fully tempered glass [۱۴].

۳.۶.۱.۳ Advantages and disadvantages of Heat Treated Glasses

The following advantages and disadvantages of heat treated glasses are important from the subject point of view:

۱. More resistance to breakage as a result of thermal stresses.

- ϣ. When it is broken, it breaks into small pieces in comparison to the large shred of annealed glass [13].
- ϣ. It is considered as a safety glazing material under the criteria of American National Standard Institute for Safety Glazing Materials Used in Buildings (ANSI Z97.1) [41].
- ξ. Heat treated glass (heat strengthened or tempered) has an optical phenomenon that is called strain pattern or quench pattern. This phenomenon appears as faint spots, blotches or lines. Strain pattern is not normally visible, but it may become apparent under certain conditions of illuminations, especially when light is polarized. The colors of the strain pattern are sometimes referred to as iridescent [44].
- ο. Neither heat strengthened nor tempered glasses can be cut or re-sized [12].
- ϧ. Strengthened and fully tempered glasses have lower resistance to scratches and abrasion than annealed glass [13].
- ϣ. Surface compression stresses higher than 140 MPa could cause the glass product to fail spontaneously [30].
- λ. The lack of symmetry in achievable glass cooling surface, glass tube, containers and other complex-shaped products can not be meaningfully tempered by thermal means [30].
- ϑ. Heat treated and tempered glasses are expensive to produce versus to annealed glass [12].

3.6.2 Strengthening by Ionic Exchange

It is based on introducing large ions in the surface of glass substituting small ions in the surface. The result is an enlarging of surface dimensions. The inner atoms will attract surface atoms preventing them to enlarge. This action will cause compression in surface layer cause in strengthening of glass. [40].

3.7 Etching

In general, etching refers to the remove of surface layers that contain the flaws by chemical action. There are two kinds of etching either isotropic or anisotropic depending upon vertical and lateral etching rates. Wet chemical processes result in isotropic etching where both the vertical and lateral etching rates are comparable, whereas dry etching process like plasma etching and reactive ion etching are anisotropic as shown in Figure (3-8) [46]. Wet chemical etching involves three essential steps [47]:

1. Transportation of reactants to the reacting surface(e.g. by diffusion).
2. Chemical reactions at the surface.
3. Transportation of the products from the surface (e.g. by diffusion).

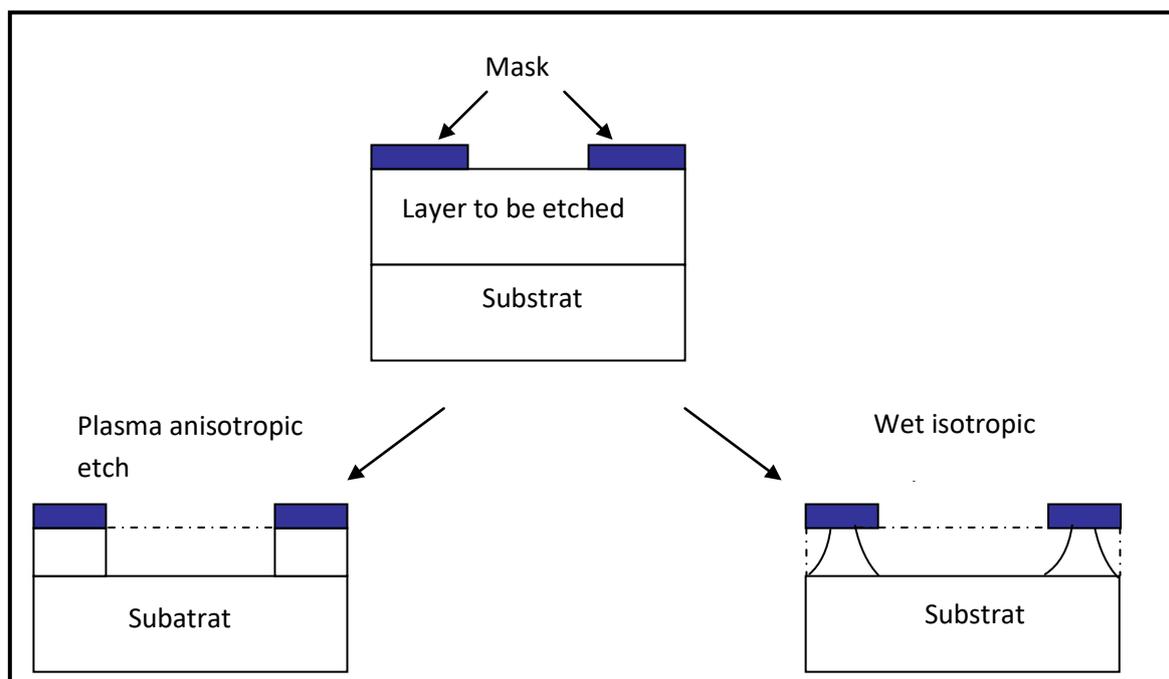
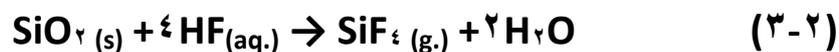


Fig.(3-8) Difference between anisotropic and wet isotropic etching [47].

3.7.1 Glass Etching

Glass is very sensitive to flaws which are formed during physical contact and during cleaning glass surfaces. Chemical etching is one of the technique that is used to remove these flaws from glass surfaces where it decreases the density of flaws on the glass surfaces [4].

Many etchants can be used to etch glass depending on its composition such as HF, Buffered HF, NaOH, NH₄OH ...etc.. After etching, the glass surface composition can alter from that of bulk depending on effectiveness of the etchants where some processes such as adsorption (e.g. F⁻ or NH₄⁺) or ion-exchange(e.g. Na⁺ for Ca⁺) can occur to varying degrees during etching [47]. If the etching bath is diluted hydrofluoric acid (HF), the chemical reaction of HF acid with silica will be [46]:



The controllable parameters in wet-etching are [47]:

1. Time.
2. Temperature.
3. Solution concentration.
4. Solution recirculation.

Recirculation and agitation are variables that produce significant changes in the etch rate. It increases the etch rate by 20%. Also the etching rate increases with temperature increasing as illustrated in Figure (3-9) [19, 47]. It is

important to note that the conventional chemical etching produce roughness that will increase with the thickness of the layer removed as shown in Figure (3-10) [48].

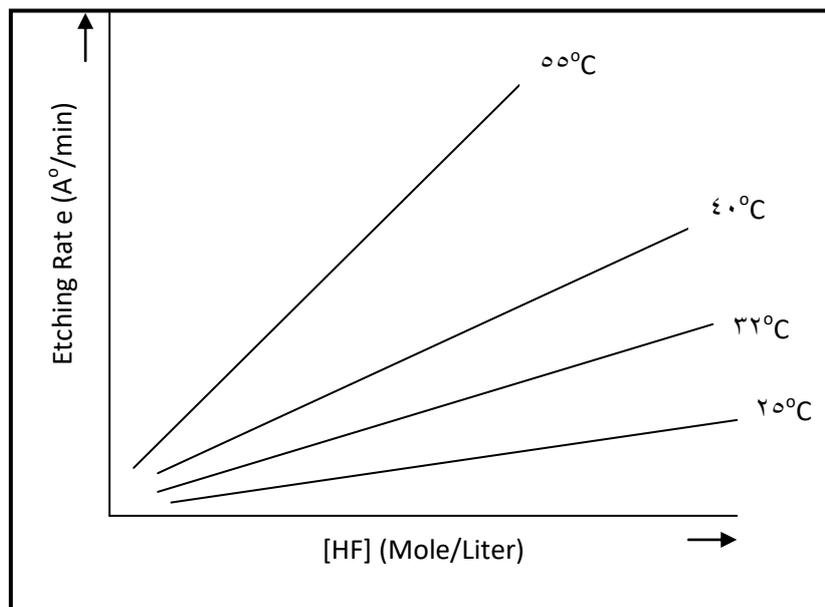


Fig.(3-9) Dependence of etching rate on temperature[46].

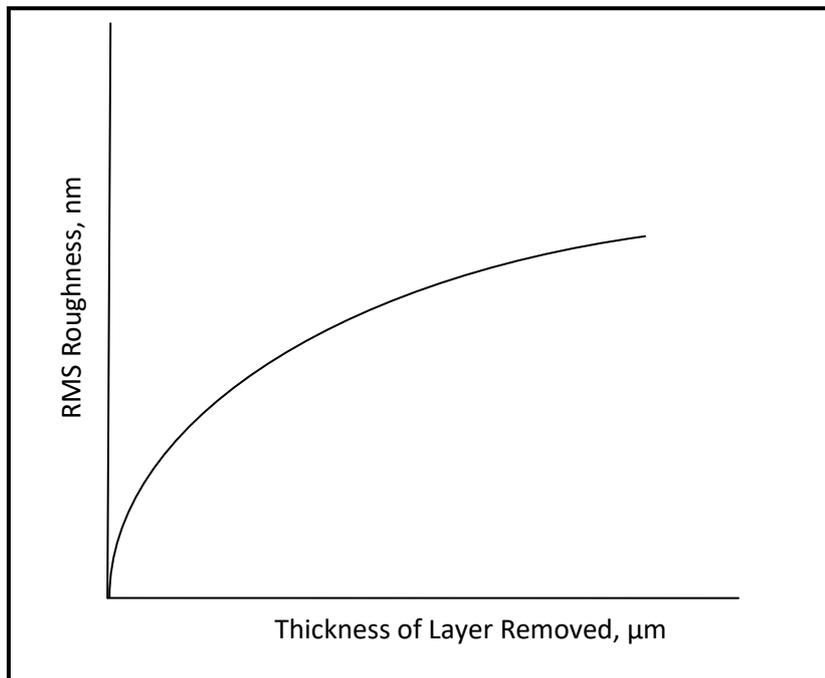


Fig.(3-10) Increasing roughness with increasing thickness of layer removed for soda-lime glass [48].

3.8 Laminating of Glass

It is a technique that has been used to improve fracture control of glass to increase the safety by avoiding spalling of glass fragments after failure has

started. Normal glass with a certain thickness has a definite strength value. Increasing the thickness of glass will aid to increase the strength. On the other hand glass is brittle and has small values of toughness, which raise the dangerous of fragmentation by damage. Laminating glass with transparent and tough material will serve as a material with adequate strength and toughness, which will be safe for human use and Figure (3-11) shows laminated glass [4].



Fig.(3-11) Schematic for laminated glass [4].

The crack bridging and the crack arrest means that the material (laminated glass) has potentially residual strength to allow to carry loads after accidental overloading which introduces new cracks. Adhesive material plays an important role in the performance of this material. When the adhesive is too strong or too thick the energy will be focused in a small region of polymer

adjacent to the actual crack. This means that infinite strain will be focused on a small area of polymer adjacent to the actual crack, which leads to failure of the material. If the adhesive fails locally, local debonding becomes possible allowing the distribution of the strain across a greater volume. It is clear, that a brittle and strong adhesive in a thin layer is required [49].

3.9 Transparent Armors

Transparent armor can be defined as the materials that have dual properties of being visually transparent and resistant to penetration by high energy projectiles and fragments. Transparent materials are an effective part of protection systems. Selecting the most appropriate glazing system is a complex process and requires understanding of the type and level of protection desired [39].

3.9.1 Normal Transparent Shields

Many years architects have been fascinated by transparency in buildings. Recent decades have shown an increase in the use of glass in facades, roofs and other structures. In normal life, safety means are taken. One of these means is the use transparent shields. The simple scheme of these shields is shown in Figure (3-12). These shields have multi applications which can be covered in the following points [50]:

1. **Resists intense impact**: During windstorm and hurricanes, windows may be subjected to gusting winds especially at coasts. Through the winds there are windborne debris and the impact of these debris can destroy normal

windows. Using these shields can be sustained winds and protect the occupants from the injuries.

۲. **Fading**: These shields protect the furniture and other materials that fade and become discolored when faced the solar energy. They can block over ۹۵% of UV energy while allowing most of visible light to pass through.

۳. **Acoustic insulation**: They correspond a super sound-damping in the place of the high noise especially in areas near airports and can be used in automobiles and trains.

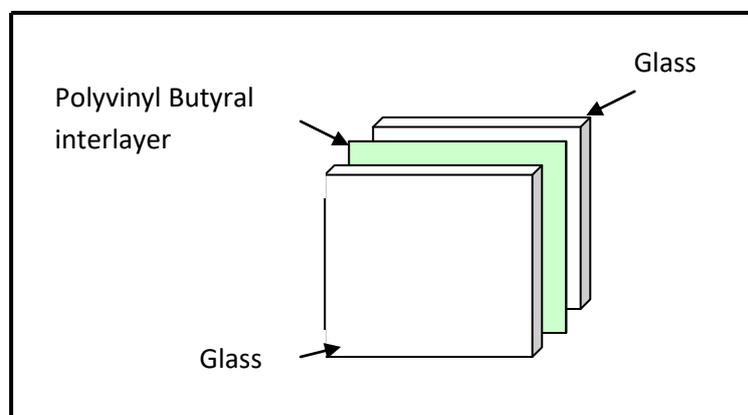


Fig.(۳-۱۲) Schematic of normal transparent shields [۵۰].

The maximum size of these shields is ۷۲ x ۱۲۰ inches depending on the interlayer material [۵۰]. The behavior of laminated glass is different than that of annealed and thermally toughened glass. When laminated glass is broken, the vinyl interlayer remains in the frame with adhering the glass fragments with the interlayer. This provides a strong barrier against forced entry. It can not be cut from one side only, which renders glasscutters. In automobile the

windshields are usually relatively simple in shape due to the requirements for a high level of optical integrity [10]. All glazing, whether automotive or architectural, require precise control of stress levels to ensure the product to meet regulatory or environmental/robustness requirements [11].

3.9.2 Ballistic Transparent Armors

Bullet-proof glass is one of these materials which is in saloon cars and in tanks etc. and it is used to protect occupants from primarily small arms fire. This type of glass has a complex structure and simply consisting of several layers of soda-lime float glass bonded to layers of polycarbonate or acrylic by urethane or by Polyvinyl Buteral (PVB) to produce a glass laminate. The primary requirements for a transparent armor system is to provide a multi-hits defeat capability while retaining visibility in the surrounding areas [12,13]. Transparent armors constructions can be classified as follows [14,15]:

1. **Glass-clad plastic**, which is composite glazing made by either sandwiching plastic sheet between laminations of glass with Polyvinyl Butyral (PVB), or Urethanes; or by laminating a plastic sheet to the back side of laminated glass to produce forced entry resistance or to be as anti-spall shield. The glass facing will exhibit cracks and loses some partial of glass by an attack.
2. **All plastic laminated construction**, polycarbonate and acrylic with adhesives are the most construction of these laminates. This type is used against burglar and missiles of medium speed.
3. **All glass laminated construction**, where is made by sandwiching a thinner layer of plastic (Polyvinyl Butyral or similar bonding agent) between two or more layer of glass. The thickness of this glass either thin, medium (1.25") or higher depending on the security level.

Annealed glass is usually used in this application to a lower cost and to the ability of produce curved products. Chemically or thermally toughened glass can be used in this field for additional strength and for their fracture shape. Other type of glass is used such as Vycor™ which is a 96% fused silica [102]. Using annealed glass or toughened glass will represent a difference of only 3% to 4% of the composite strength of the product. The type of glass selected will most often be determined by breakage characteristics, trade and manufacturing considerations [100]. Acrylic (PMMA) and polycarbonate (PC) are the most plastic sheets that are used to manufacture laminated glass. Polycarbonate has outstanding impact toughness particularly at low temperatures while acrylic has better hardness and environmental durability. This discrepancy between them returns to the motion ability of the main chains of polycarbonate molecules while in acrylic this motion is not prevalent. A new polyurethane (PU) with high performance can be replaced by polycarbonate and acrylic specially with monolithic transparent armors such as eye/face protection applications [102].

Laminations of glazy materials are adhered by interlayers of various chemical compositions and thicknesses. The adhesive interlayer materials for bonding glass to glass, glass to plastic or plastic to plastic shall be chemically compatible with the surfaces which are being bonded. Interlayers not only adhere one lamination to another but actually impart significant additional strength to the product by shock impact absorption. The interlayer also serves to retard further breakage after an adjacent lamination has shattered [103, 104].

Spallation is fracture away from the impact point caused by dynamic tensile stresses which arise from interactions of the initial, compressive stress pulse with unloading waves from the free surface [106]. The backing layer,

which is usually polycarbonate, is used to prevent the spall at the rear face of the target while acrylic has a spall behavior during impact, so it is not used as a back layer [29].

CHAPTER FOUR

EXPERIMENTAL WORK

4.1 Introduction

This chapter deals with intimate practical techniques for glass strengthening. In order to achieve this purpose, we shall apply etching and heat treatment for soda-lime glass. Many apparatus and equipments are used here to test and examine the mechanical properties of strengthened glasses.

4.2 Used Materials

4.2.1 Soda-Lime Glass

A commercial soda-lime glass is used in the present work for its availability and low cost. Chemical composition of this glass is shown in table (4-1). The thickness of glass is 2mm.

Table (4-1): Chemical composition of Soda-lime glass

SiO ₂ %	Na ₂ O%	CaO%	Al ₂ O ₃ %	K ₂ O%	Rest%
68.2	9.40	9.96	1.03	0.26	10.2

4.2.2 Hydrofluoric Acid

Hydrofluoric acid is one of the hydrogen halides. It is a colorless liquid with boiling point of 19.5°C [9]. The concentration of HF acid used in the present work is 40% and it is made by Quaker Chemical Company, Inc..

4.2.3 Acrylic

Acrylic is a thermoplastic material. It is known as a cast acrylic for production process. Acrylic sheets used in the present work are made by Affiliated Company. Compared with glass, acrylic sheet is soft and tough. It is not notch sensitive as glass but has a relatively notch sensitivity. It has resistant to organic and inorganic chemicals and outdoor weathering [10].

4.2.4 Epoxy Resin

The epoxy used in all tests is produced by LEYCO CHEM. LEYDE GMBH. It is colorless resin which have the properties given in table (4-2).

Table (4-2) The properties of epoxy [9].

Density at (23°C)	1.15 g/cm^3
Fully cured (23°C)	7 days
Compressive strength	$85-100 \text{ MPa}$
Modulus of elasticity	2800 MPa
Minimum curing temperature	10°C

4.3 Apparatus and Manufactured Equipments

1. *Furnace* / For heating samples, a vertical furnace is manufactured by the researcher as shown in Figures (4-1) with a heating rate of $1^{\circ}\text{C}/\text{min}$.



Fig.(ξ-١) Electrical furnace equipment.

٧. *Air ducts* / Two type of ducts are manufactured by the researcher to cool the test samples. Small ducts as shown in Figure (ξ-٧). They constructed of two fans with ٧٨٠٠ rpm, and at the end of them nozzles which makes the air distribute on the glass faces. The final design of the two ducts are got after many experiments done by the researcher. The design is about the same design used in plants for strengthening glasses. The distance between them is ٧٠mm. Because of difficulties to control upon the air temperature, air cooling temperatures (١٧°C, ٢٠°C, and ٣٠°C) are selected depending on the ambient air temperature in Summer or Winter.

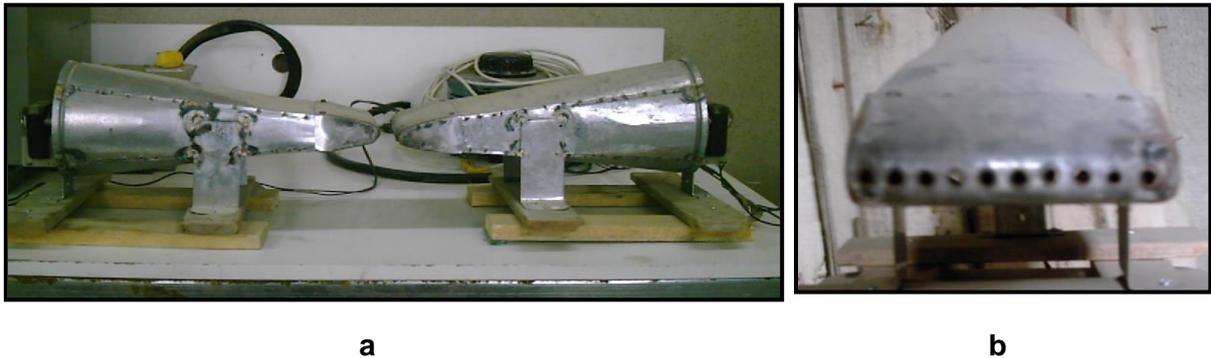


Fig.(ξ-٧) a) Ducts that are used to cool treated glass samples. b) nozzles.

Big ducts for cooling the big samples (impact samples) and they shown in Figure (ξ-٨). The distance between them is ١٠٠ mm.

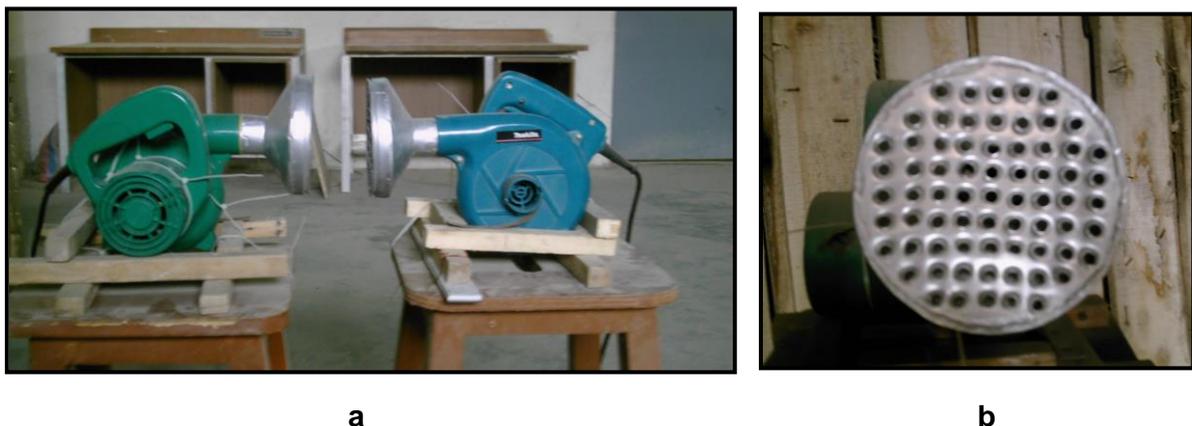


Fig.(4-3) a) Ducts that are used to cool the large impact samples. b) nozzles

3. *Microhardness Vickers apparatus.*

4. *Charpy apparatus.*

4.4 Procedures

4.4.1 Heat Treatment

This process generated compressive stresses at glass surface and tensile stresses in the glass core. The treatment demands heating and cooling the glass. There are three cooling processes techniques involved in this study which are: annealing hardening, blowing hardening, and quenching hardening.

4.4.1.1 *Annealing Hardening*

All processed samples with this technique are heated in the vertical furnace to different temperatures (e.g. 575°C, 600°C, 625°C and 650°C). After arriving each temperature, the samples are taken out from the furnace and cooled by natural air without using the ducts (free convection) to room temperature.

4.4.1.2 *Blowing Hardening*

To achieve this treatment the ducts are used in cooling process of the samples. In this technique the samples are heated to different treating

temperatures (e.g. 570°C, 600°C, 620°C and 650°C). After arriving each temperature, the samples are taking out from the furnace and are cooling by air jet that is generated by the ducts. The air jet in cooling process continues until reaching the room temperature.

4.4.1.3 Quenching Hardening

To increase cooling rate, this technique has been used in which the sample of glass is subjected to limited time of air jet and then immediately quenched in oil. The samples are heated to different treating temperatures (e.g. 570°C, 600°C, 620°C and 650°C). After arriving each temperature, the samples are taking out from the furnace and are cooling by air jet ,that is generated by the ducts, to different times (e.g. 20sec, 1.0min, 1 min, 3 min, and 5 min) and then quenched in oil at room temperature.

4.4.2 Etching

In this process, all preparation slides are cleaned in methanol alcohol. Hydrofluoric acid is diluted by distilled water to different concentrations which are (0%, 10%, 20%, 30%, and 40%). These ratios represent volume ratios. All samples mounted in a polyethylene holder are totally submerged in a polyethylene container at 30°C. After etching, the slides are washed by distilled water and methanol alcohol, then finally dried. The etching solution is recycled for each period of etching. During handling with HF acid, safety and procedural regulations is followed. Hydrofluoric acid is an extremely toxic and dangerous acid. HF should be handled in a laminar flow bench, using two pairs of neoprene gloves and eye protection. The cover of etchant bath should be tight -up.

4.4.3 Laminating Process

It is used to prepare the samples for static, dynamic, and shooting tests. In the present study, glass is combined with acrylic sheets. Because of primary adhesive materials are not available in the markets, epoxy resin is used as a glue material to tie glass and acrylic sheets together.

To produce a laminating sample, adhered material (epoxy) is poured between two or more layers of glass and acrylic. These layers are put between two wood plies. Pressure is applied on the plies to let air to escape from them and distribute the glue to whole area of the samples as shown in Figure (4-4). Adhered epoxy material is kept on for 7 days at room temperature to cure the whole laminated sample. The design of glass is called glass-clad plastic. The dimensions of the laminated samples are changed according to test. The whole thickness is varied between 12mm to 49mm according to test requirements.

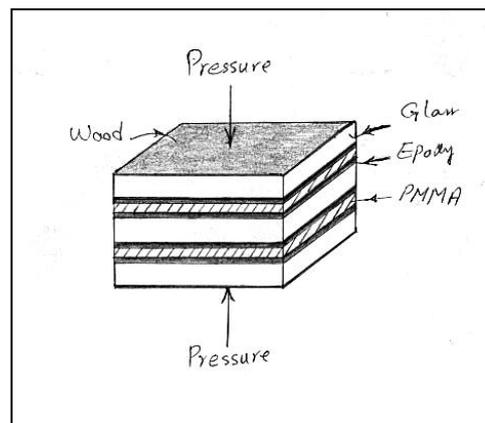


Fig.(4-4) Schematic of the forming of laminated glass.

4.5 Tests

4.5.1 Mechanical Tests

4.5.1.1 Flexure Strength:

Flexural test is used for determining the strength and deformation properties of the glass specimen under flexural three-point loading. Flexural load is the flexural stress at maximum load (according to JISR 16.1, 1981). PHYWE machine model 17071-93, Germany, was used for determination of flexural strength. The samples of soda-lime glass used in three point bending test are cut from an annealed 6mm thick soda-lime glass sheet by using a diamond saw. According to machine standard, the glass specimen of 120x20x6mm dimensions is supported on curved edge over a suitable span, and a direct load is applied at the mid point between the supports at a uniform rate of loading until breaking occurs. The loading rate was (6mm/min), and the distance between two support point (span length) was 100mm as shown in Figure (4-5). When the instrument is turned, the glass specimen will be forced by upper point crosshead, hence the specimen is trained gradually until breakage occurs. The fractural strength of three points was calculated from measured value for individual test piece, a follows [10]:

$$\text{MOR or } \sigma = \frac{3pl}{2WT^2} \quad (4-1)$$

Where:

MOR= Modulus of rupture (MPa).

σ = Three point flexural strength (MPa) or Modulus of Rupture.

l = Distance between two supporting points (mm).

p = Maximum load at breaking of tested specimen (KN).

W = Width of tested specimen (mm).

T = Thickness of tested specimen (mm).

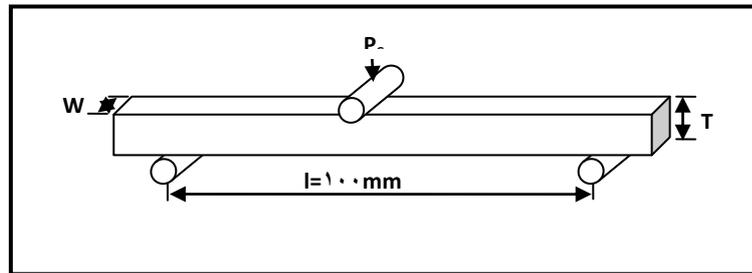


Fig.(4-5) Layout of three point flexure strength test.

4.5.1.2 Dynamic Toughness

This test can be done according to (ANSI Z97.1, 1975). In this study, Charpy apparatus for impact is used to know the absorbed energy because there is no ability to processed large samples. A rectangular samples of 100 mm length and 20 mm width are tested in this apparatus. The thickness for all samples is unlimited and is ranging from 0 mm to 49 mm. This apparatus consists of an arm with a hammer at the end. The hammer falls from a height of 1.0 m to hit the sample.

4.5.1.3 Microhardness

This test was carried out by using digital microhardness Vickers apparatus type (HVN-1000/ZBY337-80) with a diamond indenter. This instruments uses a

square-based diamond indenter with angle of 136° between the opposite faces.

A pigment is put on the glass surface to show the trace. The hardness number has been read from special table which contains the average diagonal and HVN number. The applied load was 10 N with loading duration of 10 Second. The Vickers microhardness (HVN) was calculated using the following equation:

$$\text{HVN} = 1.8544 \frac{P}{D_v^2} \quad \text{in (Kgf/mm}^2\text{)} \quad (4-2)$$

where:

P = applied load (Kgf).

D_v = mean diagonal of the indentation (μm).

4.5.1.4 Shooting

The ballistic test is performed according to the National Institute of Justice (NIJ) standards (NIJ Standard 108.1). This standard was chosen because it enables the user to gain an understanding of the aspects of how to improve the ballistic impact resistance of various systems without requiring a large amount of costly test samples. Figure (4-6) shows the layout of shooting process following the NIJ standard. According to this standard, when a projectile penetrates the specimen, if any fragment of the projectile or specimen creates a hole in the aluminum witness plate then it is considered as a complete penetration. If no hole exists, it is considered as a partial penetration [66].

The impact samples have dimensions of $120 \times 120 \times 0$ mm. The thickness of these samples is ranging from 16 to 49 mm after lamination with acrylic layers. The test specimen is rigidly clamped between two steel frames. A gasket of rubber is located between glass and steel to avoid any fracture of glass due to friction between glass and steel. A plate of 1.0 mm thick aluminum located behind the specimen at a distance of 10.24 cm to record penetration of samples as shown in Figure (4-7). The weapon used in this test was a Kalashnikov rifle [66].

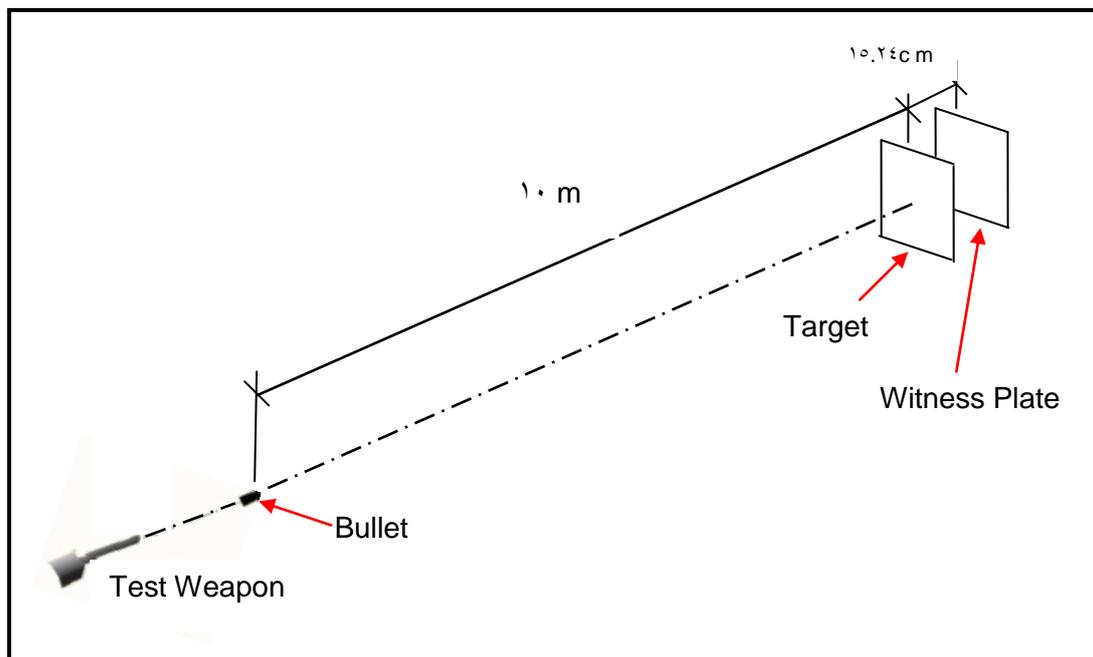


Fig.(4-6) Layout of the shooting process.

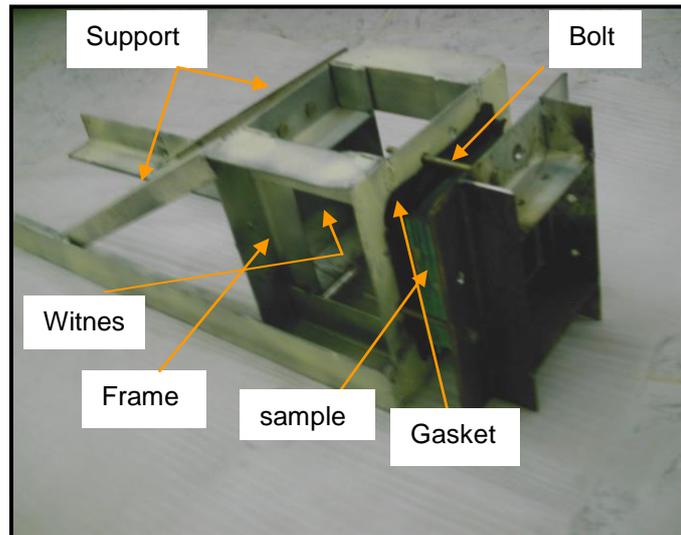


Fig.(4-7) Holder of impact samples.

4.5.2 Optical

It is an optical apparatus which is used to show the strain pattern that is formed in the heat treated glass. It contains a fluorescent in a special shape and filter. The angle between the fluorescent and the filter is 90° to obtain the strain pattern. In this examination, red color indicates that the stresses are compression and the blue color is tension.

CHAPTER FIVE

RESULTS AND DISCUSSION

5.1 Introduction:

This chapter includes the main results which are obtained from the experimental observations. It presents the effect of strengthening methods on the strength of annealed soda-lime glass. To arrive the goal of this study which is developing the impact strength in glass. Several tests are done to know the best conditions for strengthening glass against projectiles. The tests include modulus of rupture, hardness, dynamic impact and shooting by rifle.

0.2 Modulus of Rupture

This test is commonly carried out on glass in which it gives an indication of glass strengthening. The behavior of all curves of heat treatments are matched with the Hélène carré and Laurent Daudeville where they have mentioned that the residual compressive stresses grew with initial treating temperature to 600°C and stay constant beyond it up to 680°C [12]. Each point in these Figures represents average of three values.

0.2.1 Annealing Hardening

Figure (0-1) presents the effect of initial treating temperatures on the fracture strength of glass with different indoor air temperatures. It is clear that the strength of as received glass is (64.8Mpa). Above glass transition temperature (T_g) of soda-lime glass ($574^{\circ}\text{C}-520^{\circ}\text{C}$), the strength increases with increasing initial treating temperature until 600°C . The main reason of this is due to the increasing in the residual compressive stresses in the surfaces of glass. After the temperature (600°C) the glass begins in distortion so, that it is impossible to continue in heating [12].

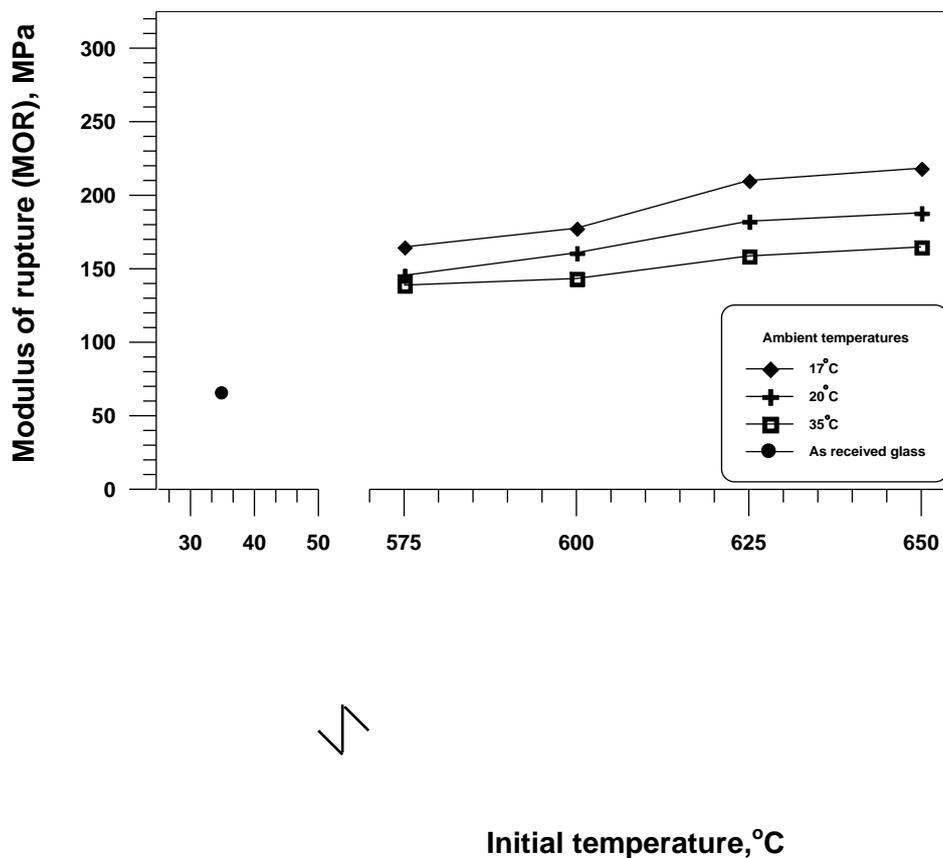


Fig. (0-1) Fracture strength versus initial treating temperatures of glass without fan cooling.

0.2.2 Blowing Hardening

Figure (0-2) shows the effect of initial treating temperatures on the fracture strength of glass with different air blowing temperatures. It indicates an increase in fracture strength of glass with increasing initial treating temperature. The cooling process of individual treated temperatures were achieved with air blowing at three temperatures which were (17°C, 20°C, and 35°C). The air blowing temperature of 17°C gives the best and highest values of strengthening for all initial treating temperatures of blown glass.

Fracture strength of thermally treated glass has been affected by the temperature of air cooling. It is higher at lower air temperature (17°C) because of the increase in cooling rate and due to the increase in thermal gradient through thickness of glass. This gradient causes a residual stresses on each side of glass [62].

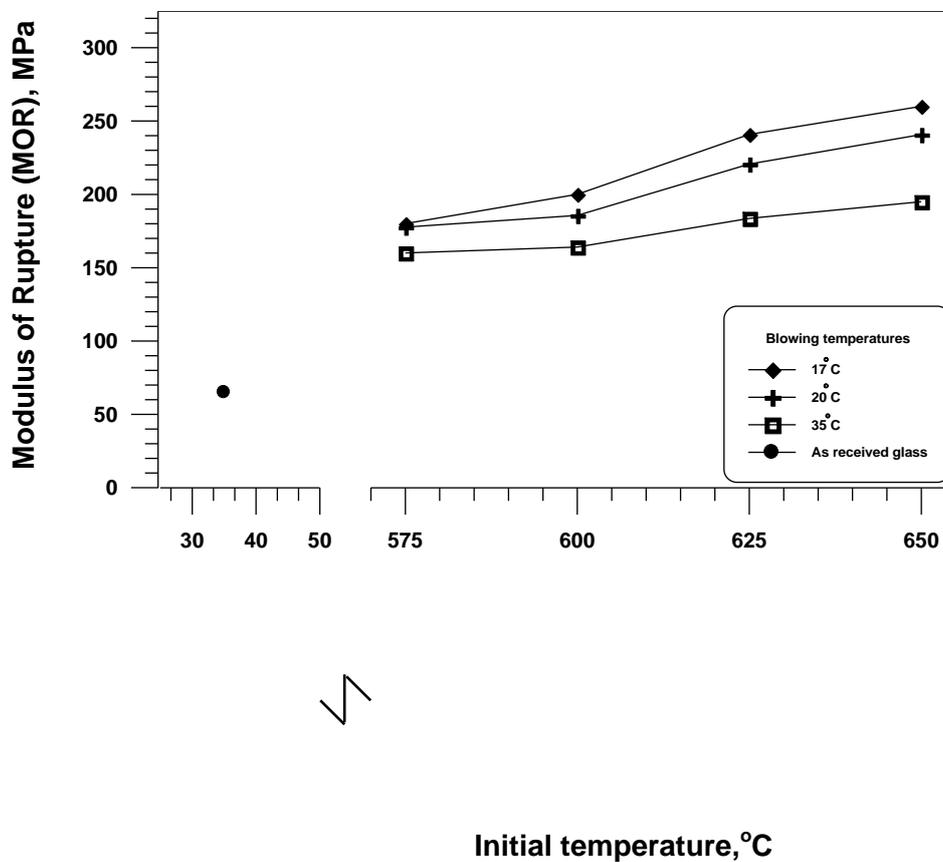


Fig. (2-2) Fracture strength versus initial treating temperatures of glass with fan cooling.

In both Figures (2-1) and (2-2) the increase of strength is compatible with the built up residual stresses. Cooling speed plays an important role in the

fracture strength of treated glass. As shown in Figure (٥-٢) the cooling is done by fans (air jet) while in Figure (٥-١) is without fans. Generally, the fracture strength of treated glass by air blowing hardening is more than that of annealed hardening. The results of Figures (٥-١) and (٥-٢) are given in table (٥-١).

For better comparison between heat treatments of samples with and without using fan cooling, table (٥-١) shows the modulus of rupture of both producers. It is clear that the heat treatment with fan gives higher values of modulus of rupture. In general, the increasing of temperature difference between glass and air will give a higher value fracture strength. This fact belongs to the increase of hardened thickness, which gives more strength [٦٢].

Table (٥-١) Modulus of rupture (MOR) results of heat treated glass with different cooling speed

Temperature of cooling air, °C	Initial temp., °C	Without fans				With fans			
		٥٧٥	٦٠٠	٦٢٥	٦٥٠	٥٧٥	٦٠٠	٦٢٥	٦٥٠
١٧	MOR	١٦٤.٨	١٧٧.٥	٢١٠	٢١٨.٣	١٨٠	٢٠٠	٢٤٠.٨	٢٦٠
٢٠	MOR	١٤٥.٤	١٦٠.٨	١٨٢.٣	١٨٨	١٧٧.٦	١٨٥.٦	٢٢٠.٦	٢٤٠.٧
٣٥	MOR	١٣٨.٨	١٤٣.٣	١٥٨.٦	١٦٤.٨	١٦٠	١٦٤	١٨٣.٦	١٩٥

٥.٢.٣ Quenching Hardening

Figures (٥-٣, ٥-٤, and ٥-٥) show the effect of initial treating temperatures and oil quenching on the fracture strength of glass. Cooling process in these Figures is done by using air jet to different times at the same air temperatures (١٧°C, ٢٠°C, and ٣٥°C) and then quenched in oil to cool at room temperature. As mentioned above, the increasing in fracture strength of treated glass may occur by increasing the difference of temperature between the core and the

surface of treated glass. Furthermore, one can see whenever the air jet time is low, the fracture strength of treated glass is high and vice versa. This behavior may be explained by existence of balance between air jet time and dipping in oil. The unbalance in this process, causes failure for the treated samples. This fact is clear from Figure (°-°), where we have no value of modulus of rupture by the blowing time of 20 seconds due to break of sample. Hence, a shortening in time of air cooling before oil quenching causes an increase in the modulus of rupture.

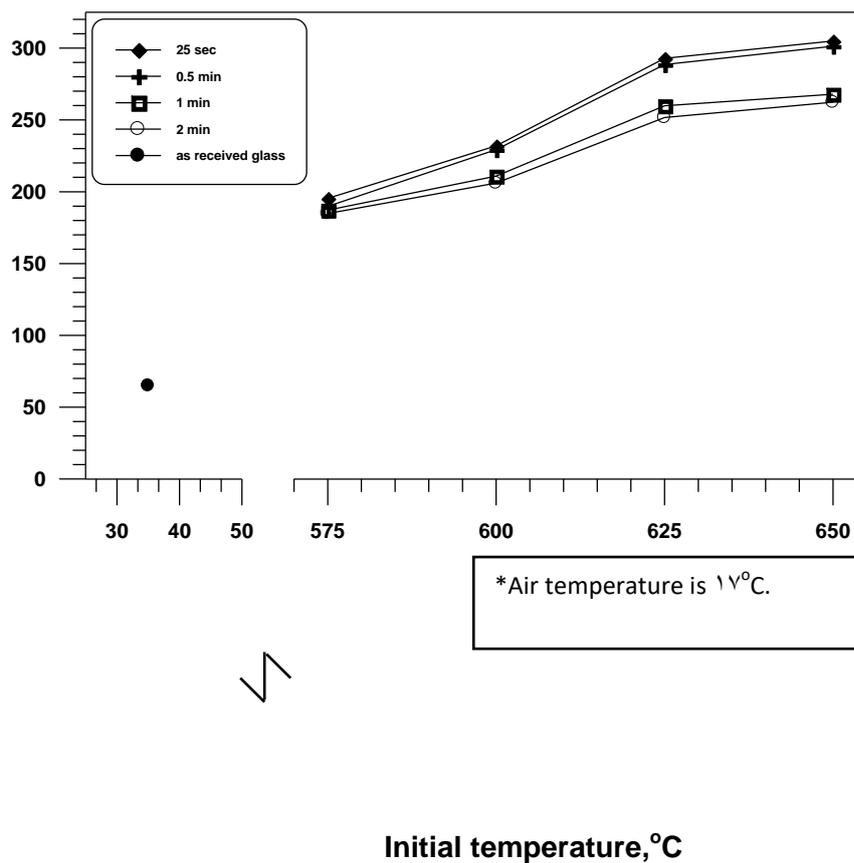


Fig. (°-°) Fracture strength of glass treated by

quenching in oil at 17°C.

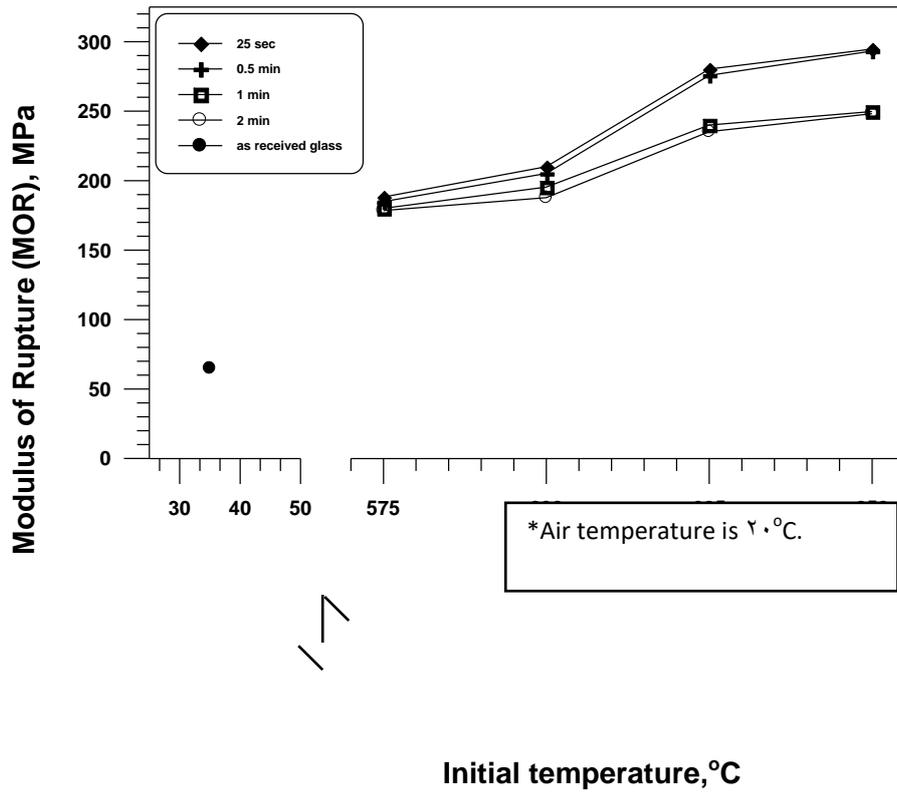
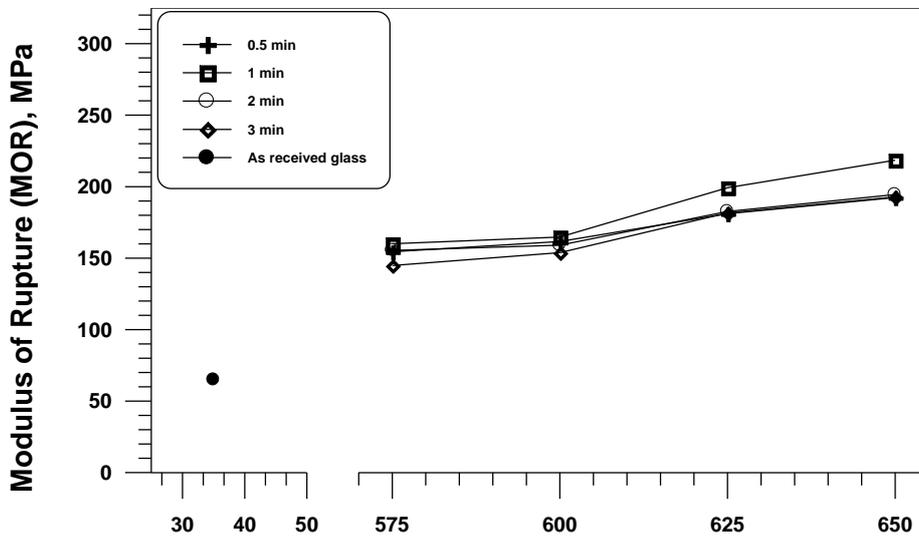


Fig. (a-c) Fracture strength of glass treated by quenching in oil at 17°C.





*Air temperature is 30°C.

Initial temperature, °C

Fig. (0-0) Fracture strength of glass treated by quenching in oil at 30°C.

Table (0-2) shows a comparison between all values of modulus of rupture for quenching with oil and quenching with air only. The quenching in oil gives higher value as in air. The strength is increasing by increasing the quenching temperature difference, which causes building of harder glass surface.

Table (٥-٢) Results of modulus of rupture of heat treated glass with different times of air blowing

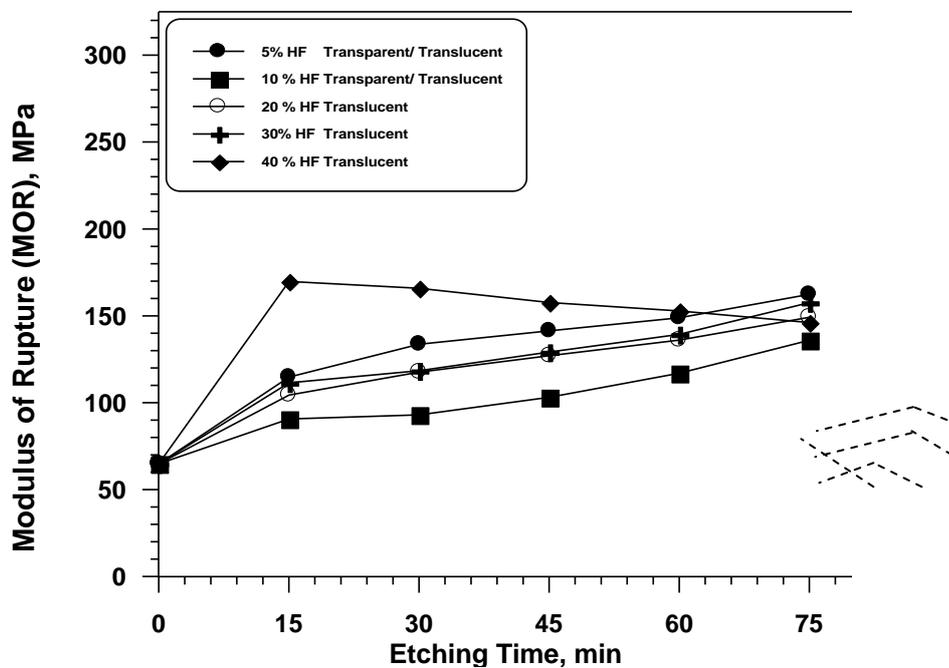
Initial treating Temperature, °C	Temperature of cooling air, °C	Time of air blowing, min (then quenched in oil)					Cooling with fan	
		٢٥sec.	٠.٥	١	٢	٣		
٥٧٥	١٧	MOR	١٩٥.٣	١٨٩.٧	١٨٧.٢	١٨٤.٨	-	١٨٠
٦٠٠	-	MOR	٢٣١.٩	٢٢٩.٣	٢١٠.٨	٢٠٦	-	٢٠٠
٦٢٥	-	MOR	٢٩٢.٨	٢٨٨.٥	٢٥٩.٨	٢٥١.٦	-	٢٤٠.٨
٦٥٠	-	MOR	٣٠٤.٩	٣٠١.٣	٢٦٧.٩	٢٦٢.٣	-	٢٦٠
٥٧٥	٢٠	MOR	١٨٨.٢	١٨٤.٩	١٨٠	١٧٨.٤	-	١٧٧.٦
٦٠٠	-	MOR	٢١٠	٢٠٥.١	١٩٥.٣	١٨٧.٧	-	١٨٥.٦
٦٢٥	-	MOR	٢٨٠.٣	٢٧٥.٨	٢٤٠	٢٣٥.٢	-	٢٢٠
٦٥٠	-	MOR	٢٩٤.٧	٢٩٣.٢	٢٤٩.٧	٢٤٨.٢	-	٢٤٠.٧
٥٧٥	٣٥	MOR	-	١٥٤.٤	١٦٠	١٥٥.٣	١٤٤.٨	١٦٠
٦٠٠	-	MOR	-	١٦١.٦	١٦٤.٧	١٥٩.٢	١٥٣.٨	١٦٤
٦٢٥	-	MOR	-	١٨١.٦	١٩٩.٢	١٨٢.٦	١٨١.٦	١٨٣.٦
٦٥٠	-	MOR	-	١٩٢.٣	٢١٨.٤	١٩٤.٤	١٩٢.٨	١٩٥

٥.٢.٤ Effect of Etching

Figure (٥-٦) illustrates the effect of etching time on the fracture strength of glass using different concentrations of Hydrofluoric acid (HF). All the specimens treated with different concentrations of Hydrofluoric acid show increasing in their fracture strength. This is because of decreasing in surface flaws density and etching process which blunts the crack tips [١٦]. In low concentration of (HF) acid, glass specimen needs more time to reach its maximum fracture strength. Also, this process of etching removes small layers of glass surface and causes a thin smooth surface of glass, which shows transparency up to its

maximum fracture strength. After this stage, the surface of glass will acquire some roughness and pits with large diameter. This is a characteristic feature of translucent appearance of glass. On the other hand, the high concentration of (HF) acid will cause reducing in time to reach its maximum fractural strength, and the transparency appearance will be early absent, since more thickness of glass layer will be removed. So, 5% and 10% (HF) for 1.20 hour are considered the best result of glass strengthening, where the fracture strength was 162.2 and 136.4MPa respectively. The specimens have been treated with 5% and 10% (HF) will be adapted for all next tests.

The behavior of results of the etching process comes with agreement of researcher [17].



Etching time, min

Fig. (2-6) Effect the etching time on the fracture strength of glass.

2.2.2 Etching and Quenching hardening

As discussed in article 2.2.3 that the best results of quenching process were by using jet air for 30 seconds within ambient temperature of 17°C, then quenching in oil. On the other hand, etching process has been adopted of 0% and 10% HF acid for 1.20 hour.

Figure (2-7) shows the fracture strength of initial treating temperature glass when it is subjected to both quenching and etching (0% HF) processes. There is gradually an increase in fracture strength.

Figure (2-8) shows the fracture strength of initial treating temperatures glass when it is subjected to both quenching and etching (10% HF) processes. According to these conditions, fracture strength of treated glass shows similar behavior of that mentioned in Figure (2-7); but the values of fracture strength are higher. This may be attributed to the accumulated effect of both etching at 10% HF (reducing flaws density and quenching (residual stresses)) [13]. It is clear from Figures (2-7) and (2-8), that the etching process does not provide any enhancement to raise the fracture strength of treated glass. For this

reason, the following glass specimen can be thermally processed only without etching, unless it demands enhancement in bonding strength when glass is used as a laminated habit.

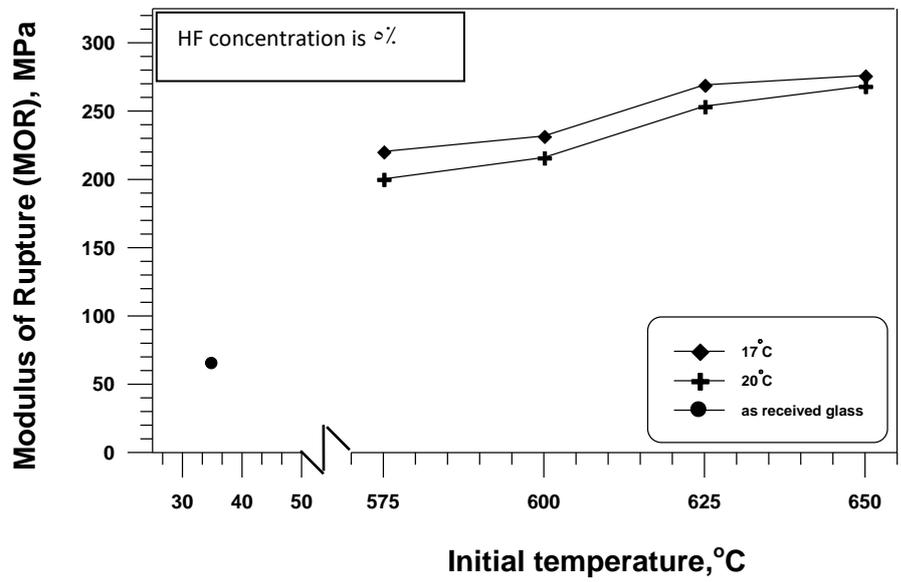
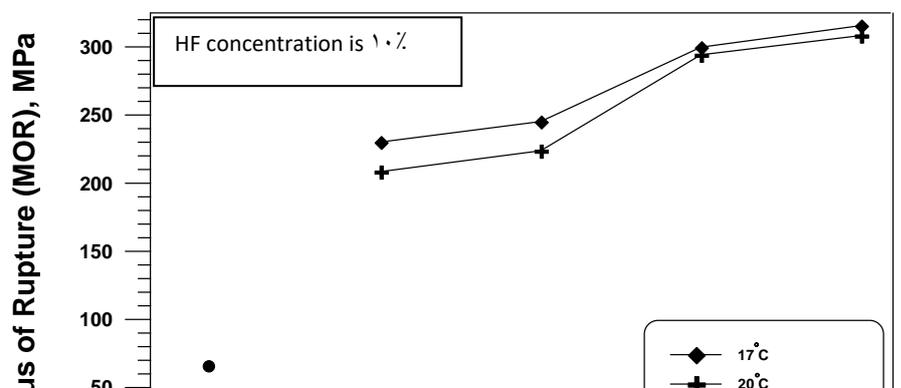


Fig. (0-1) Fracture strength of treated glass (etching of 0% HF and quenching).



**Fig. (٥-٨) Fracture strength of treated glass
(etching of ١٠٪ HF and quenching).**

Table (٥-٣) shows results of the fracture strength of glass processed with etching and quenching process.

Table (٥-٣) Fracture strength results of glass processed with etching and quenching processes.

HF concentration%	Initial treating Temperature, °C	Temperature of air cooling, °C	Air blowing time, min	
				٢٥sec.
٥	٥٧٥	١٧	MOR	٢٢٠.٤
	٦٠٠	-	MOR	٢٣١.٧
	٦٢٥	-	MOR	٢٦٩.٢
	٦٥٠	-	MOR	٢٧٦
	٥٧٥	٢٠	MOR	٢٠٠.٣
	٦٠٠	-	MOR	٢١٦
	٦٢٥	-	MOR	٢٥٣.٦
	٦٥٠	-	MOR	٢٦٨.٣
١٠	٥٧٥	١٧	MOR	٢٣٠.٢
	٦٠٠	-	MOR	٢٤٥.٢
	٦٢٥	-	MOR	٢٩٩.٨
	٦٥٠	-	MOR	٣١٥.٧
	٥٧٥	٢٠	MOR	٢٠٨.٤
	٦٠٠	-	MOR	٢٢٣.٨
	٦٢٥	-	MOR	٢٩٤.٢
	٦٥٠	-	MOR	٣٠٨.٣

٥.٣ Hardness

The behavior of hardness of all the following treated glasses are in agreement with that mentioned by Everstiejin et al [٢٨].

٥.٣.١ Annealing Hardening

Figure (٥-٩) reveals the effect of initial treating temperature on the Vickers hardness number of heat treated glass. It shows that the glass hardness decreases with increasing initial treating temperature.

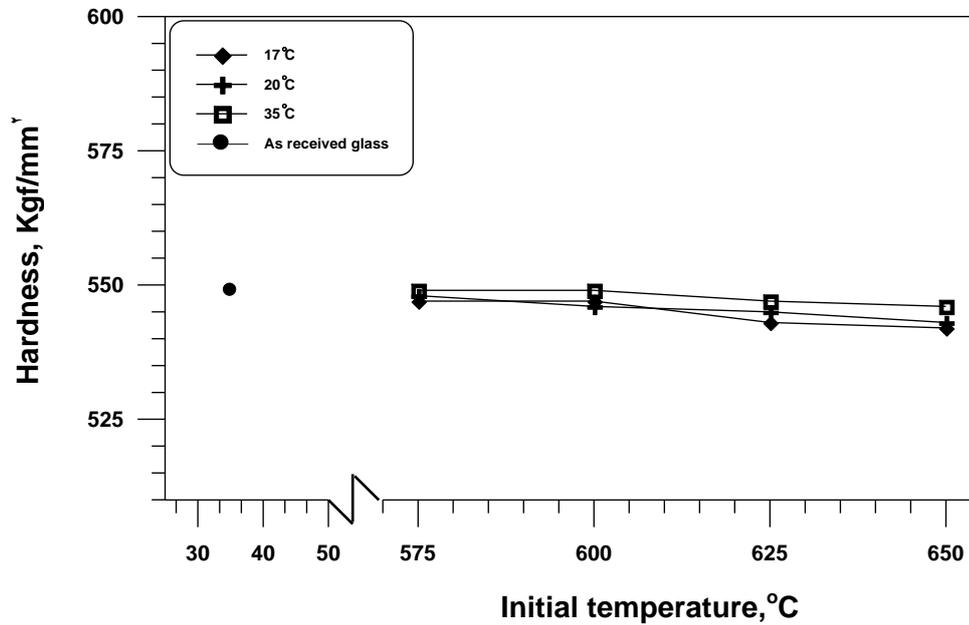


Fig. (٥-٩) Vickers hardness numbers of heat treated glass without fan quenching

٥.٣.٢ Blowing Hardening

Figure (٥-١٠) shows reversal relationship between heat treated glass with the air blowing effect and their Vickers hardness number. This behavior is similar to that illustrated in Figure (٥-٩). It has been observed that whenever air blowing present, the Vickers hardness number of heat treated glass is more decreased than that of natural cooling. As a result of this, the slope of Vickers

hardness number is more descending than that found in Figure (٥-٩). It is believed that when annealed glass is subjected to air blowing quench, the structure of glass will be of low density [٧٧], so according to this, its hardness tends to decrease gradually with decreasing air cooling temperatures as shown in Figures (٥-٩ & ٥-١٠). Table (٥-٤) illustrates the results of Figures(٥-٩) and (٥-١٠) for comparison.

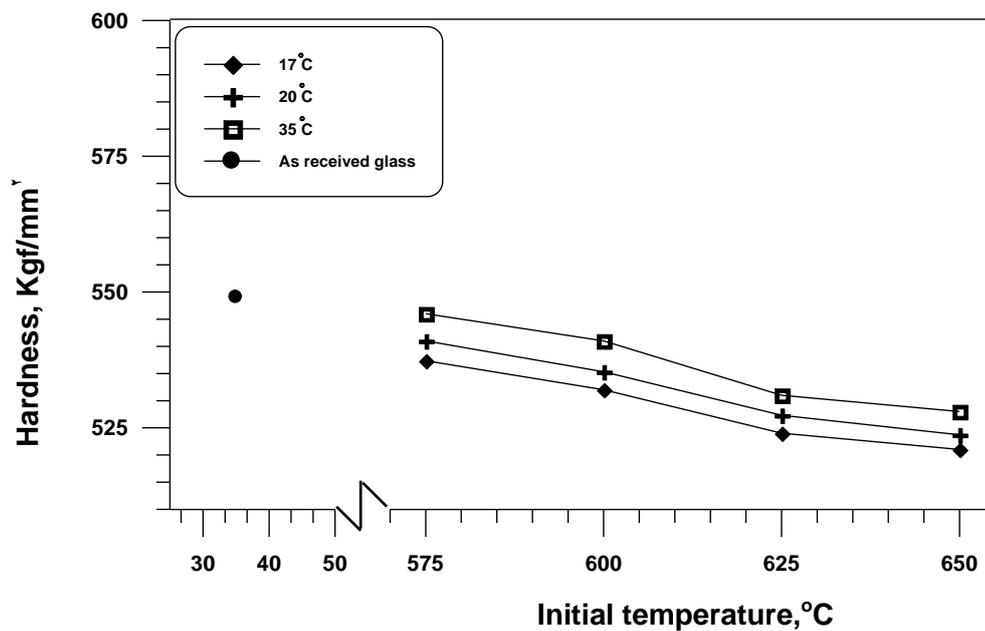


Fig. (٥-١٠) Vickers hardness numbers of heat treated glass with fan quenching.

Table (٥-٤) Vickers hardness numbers of heat treated glass with and without fan quenching.

Temperature of cooling air, °C	Initial temp., °C	Without fans				With fans			
		٥٧٥	٦٠٠	٦٢٥	٦٥٠	٥٧٥	٦٠٠	٦٢٥	٦٥٠
١٧	Hardness	٥٤٦.٣	٥٤٧	٥٤٣.٣	٥٤١.٧	٥٣٧.٣	٥٣٢	٥٢٤	٥٢١.٣
٢٠	Hardness	٥٤٨.٧	٥٤٦.٣	٥٤٤.٣	٥٤٣.٣	٥٤٣	٥٣٧.٣	٥٢٩.٣	٥٢٦.٧
٣٥	Hardness	٥٤٩	٥٤٩	٥٤٦	٥٤٤.٣	٥٤٦	٥٤٣	٥٣٢	٥٢٩.٣

٥.٣.٣ Quenching Hardening

Figures (٥-١١), (٥-١٢), and (٥-١٣) show the relationship between the initial treating temperature and Vickers hardness number of heat treated glass. All specimens in these Figures were cooled by air jet to different times then quenched in oil. The ambient temperature of cooling in Figure (٥-١١) is ١٧°C, in Figure (٥-١٢) is ٢٠°C, and in Figure (٥-١٣) is ٣٥°C. They reflected a reversal relationship, where the hardness is increased as the initial treating temperature decreased. This behavior is also established, because the surface will have lower density.

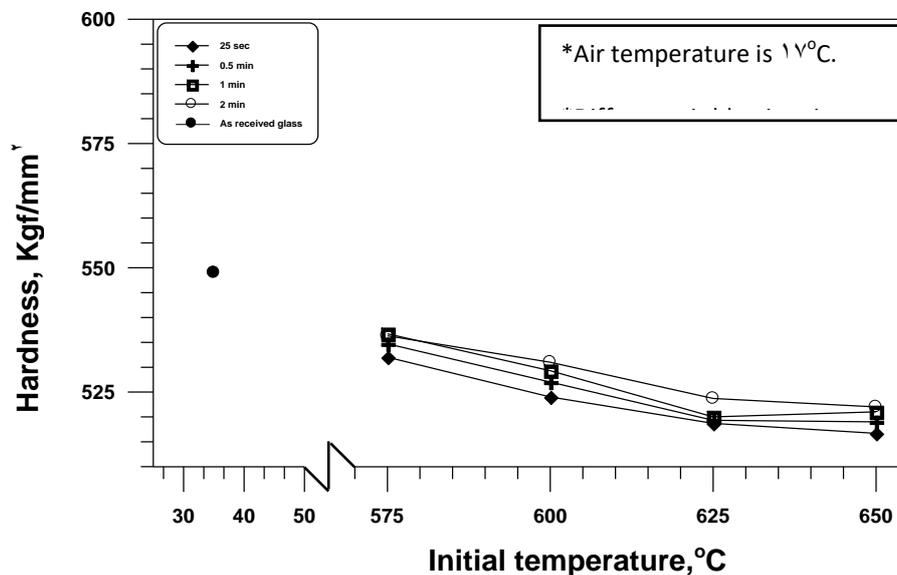


Fig. (٥-١١) Effect of initial temperatures

on the hardness of heat treated glass.

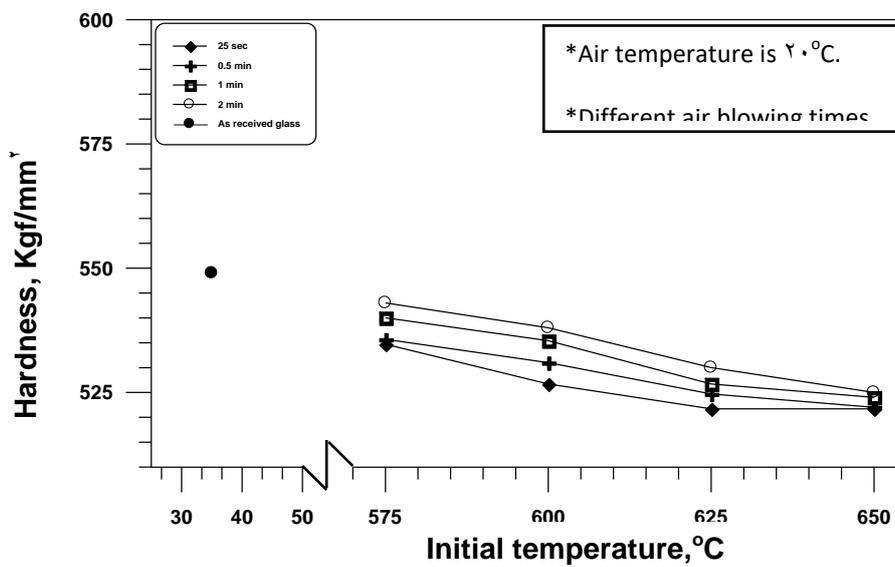


Fig. (٥-١٢) Effect of initial temperatures

on the hardness of heat treated glass

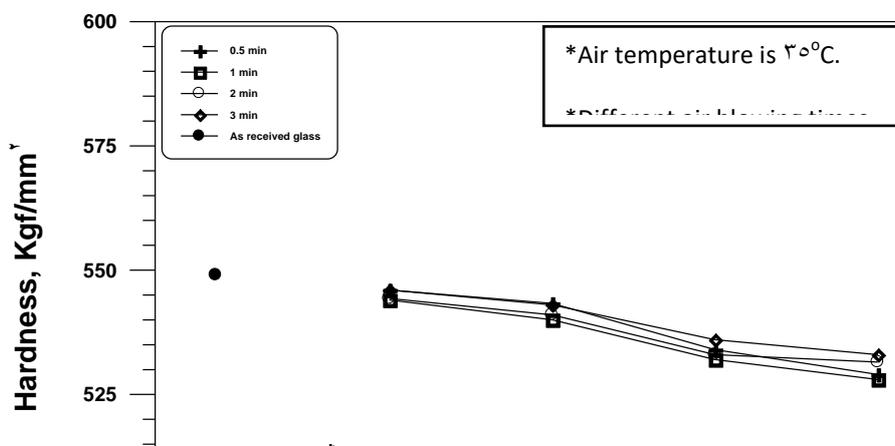


Fig. (٥-١٣) Effect of initial temperatures on the hardness of heat treated glass.

Table (٥-٥) displays the results of these Figures for comparison.

Table (٥-٥) Vickers hardness numbers of heat treated glass.

Initial treating Temperature, °C	Temperature of cooling air, °C	Time of blowing air, min					
		٢٥sec.	٠.٥	١	٢	٣	
٥٧٥	١٧	Hardness	٥٣٢	٥٣٤	٥٣٦.٧	٥٣٦.٣	-
٦٠٠	-	Hardness	٥٢٤	٥٢٧	٥٢٩.٣	٥٣١	-
٦٢٥	-	Hardness	٥١٨.٧	٥١٩.٣	٥١٩.٧	٥٢٣.٧	-
٦٥٠	-	Hardness	٥١٦.٧	٥١٩	٥٢١	٥٢٢	-
٥٧٥	٢٠	Hardness	٥٣٤.٧	٥٣٥.٧	٥٤٠	٥٤٣	-
٦٠٠	-	Hardness	٥٢٦.٧	٥٢٩.٤	٥٣٥.٤	٥٣٨	-
٦٢٥	-	Hardness	٥٢١.٧	٥٢٤	٥٢٦.٧	٥٣٠	-
٦٥٠	-	Hardness	٥٢١.٧	٥٢٢	٥٢٤	٥٢٥	-
٥٧٥	٣٥	Hardness	-	٥٤٦	٥٤٤	٥٤٤.٣	٥٤٦
٦٠٠	-	Hardness	-	٥٤٣.٣	٥٤٠	٥٤١	٥٤٣.٣
٦٢٥	-	Hardness	-	٥٣٤.٧	٥٣٢	٥٣٣.٣	٥٣٥.٣
٦٥٠	-	Hardness	-	٥٢٩.٣	٥٢٨	٥٣٢.٣	٥٣٣.٣

٥.٣.٤ Effect of Etching

Figure (٥-١٤) represents the relationship between glass etching time (minutes) and corresponding Vickers hardness number (Kgf/mm^٢). It may be noted from this Figure that, there is an increase in hardness of etched glass for both concentrations ٥% and ١٠% HF up to ٧٥ minutes. After this time of

etching, Vickers hardness number will be decreased. It is believed that, any smoothness of glass surface will cause the first on ascending order in hardness number up to 75 minutes etching time. After that time the glass surface will be probably damaged giving decreasing order in Vickers hardness number. Also, it may be noted that whenever a concentration of etching is high (10%), the corresponding Vickers hardness number is high too in comparison with that of low concentration (5%).

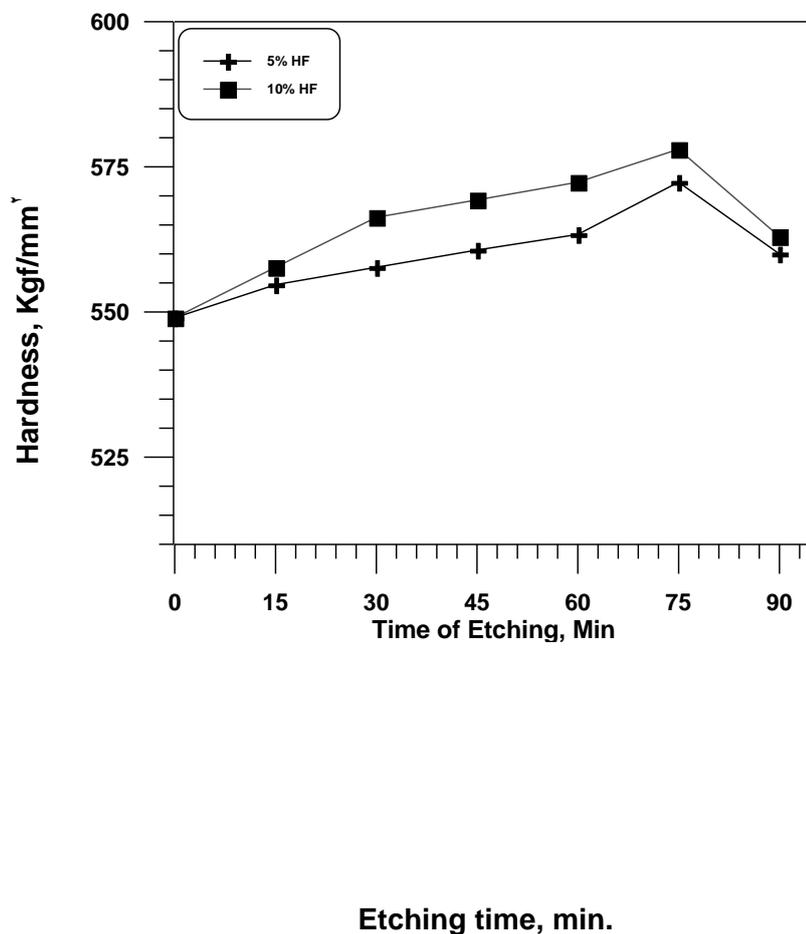


Fig. (2-14) Shows the effect of etching time on the hardness of glass.

3.3 Etching with Quenching hardening

Figures (3-10 and 3-16) show the effect of the initial treating temperature on the Vickers' hardness of etched and heat treated glass. Cooling process in these Figures is done by air blowing to 20 seconds at ambient air temperatures (17°C and 20°C) and then quenched in oil. All specimens in Figure (3-10) are etched by 5% HF while in Figure (3-16) the etching was by 10% HF. It has been observed, that the values in these Figures have the same decreasing behavior of those in Figures (3-11, 3-12, and 3-13) but somewhat higher than them. This raising in hardness value deduced from the modification in the surface state of treated glass by etching.

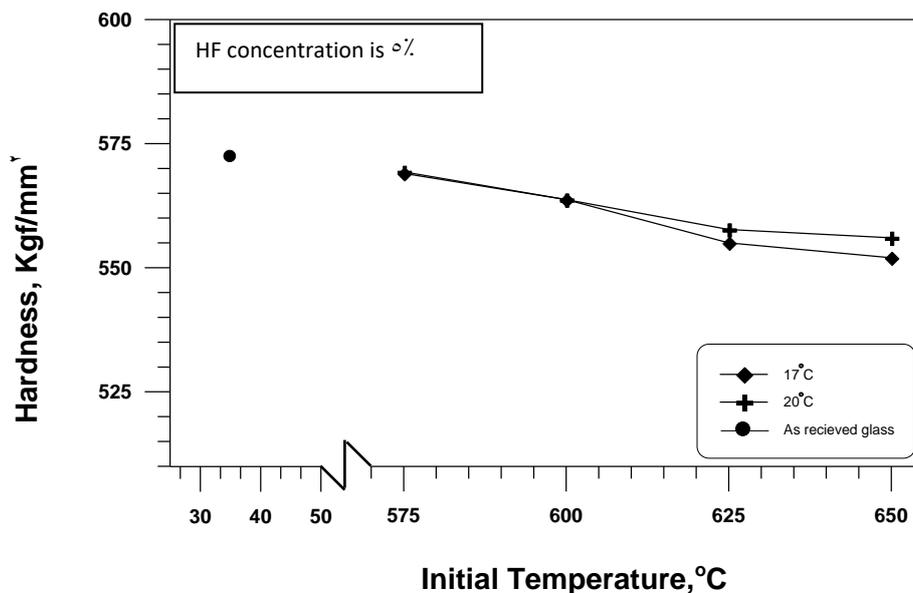


Fig. (9-15) Effect of Initial temperatures on the hardness of treated (etching (10%) and heat treatment) glass

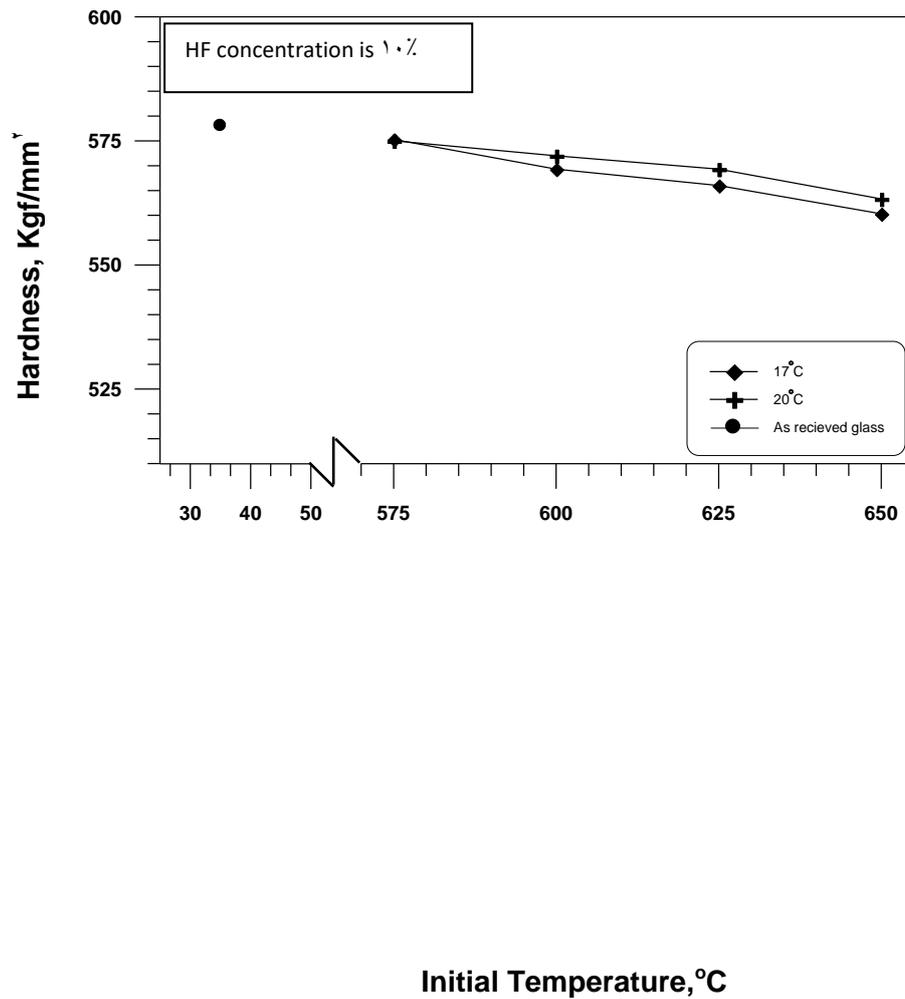


Fig. (9-16) Effect of Initial temperatures on the glass. Hardness of treated (etching (10%) and heat treatment)

Table (٥-٦) shows results of hardness numbers of heat treated and quenched glass with and without etching for comparison.

Table (٥-٦) Shows the results of etching process on hardness of heat treated glass

HF concentration%	Initial treating Temperature, °C	Temperature of cooling air, °C	Heat treated glass with etching		Heat treated glass without etching	
			Time of blowing air, min, ٢ ^o sec.	Time of blowing air, min, ٢ ^o sec.	Time of blowing air, min, ٢ ^o sec.	Time of blowing air, min, ٢ ^o sec.
٥	٥٧٥	١٧	Hardness	٥٦٩	٥٣٢	
	٦٠٠	-	Hardness	٥٦٣.٧	٥٢٤	
	٦٢٥	-	Hardness	٥٥٥	٥١٨.٧	
	٦٥٠	-	Hardness	٥٥٢	٥١٦.٧	
	٥٧٥	٢٠	Hardness	٥٦٩.٣	٥٣٤.٧	
	٦٠٠	-	Hardness	٥٦٣	٥٢٦.٧	
	٦٢٥	-	Hardness	٥٥٧.٧	٥٢١.٧	
	٦٥٠	-	Hardness	٥٥٨	٥٢١.٧	
١٠	٥٧٥	١٧	Hardness	٥٧٥.٣	٥٣٢	
	٦٠٠	-	Hardness	٥٦٩.٣	٥٢٤	
	٦٢٥	-	Hardness	٥٦٣.٣	٥١٨.٧	
	٦٥٠	-	Hardness	٥٦٠.٣	٥١٦.٧	
	٥٧٥	٢٠	Hardness	٥٧٥	٥٣٤.٧	
	٦٠٠	-	Hardness	٥٧٢	٥٢٦.٧	
	٦٢٥	-	Hardness	٥٦٩.٣	٥٢١.٧	
	٦٥٠	-	Hardness	٥٦٣.٣	٥٢١.٧	

٥.٤ Strain Pattern or Quench Pattern Photos

Strain pattern of thermally treated glass have been recorded qualitatively as photos visualized from polarscope apparatus. It may be noted from the photos, that any increase in red color in specimen is generated from compressive stresses. As a result of this, the specimen shows an iridescence. Figure (١) can not display any color because it is untreated. Figure (٢) represents the strain pattern of heat treated glass at ٦٥٠°C then cooled in air without air jet. This Figure has strain pattern different from other Figures because the hot glass surfaces is directly in contact with natural cooling air. Figures (٣, ٤, ٥, and ٦) show the strain pattern in the samples of heat treated at (٦٥٠°C), cooled with ambient temperature of (١٧°C) and by air jet to different times (٢^osec, ١.٥min, ١min, and ٢min) respectively finally quench in oil. It has been observed that all the above mentioned photos have the same

pattern because they are subjected to the same condition and manner of air cooling. They show increasing in red color which indicates increasing in compressive stresses as the temperature increase. Figure (Y) represents the impact sample where it shows the strain pattern similar to that of upper Figures. It is noted from the Figures that the strain pattern take the shape of the air blown out of the nozzles. The strain pattern presents in this study are compatible with behavior of a technical report [٤٤].



Fig. (١) strain pattern of untreated glass.

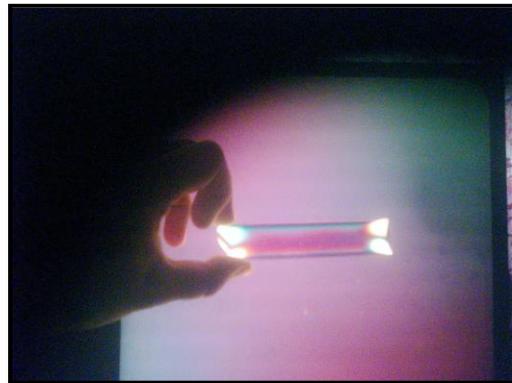


Fig. (٢) Strain pattern of heat treated glass at ٦٥٠°C then cooled in air

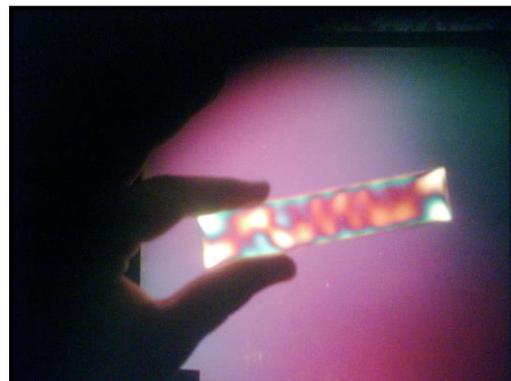
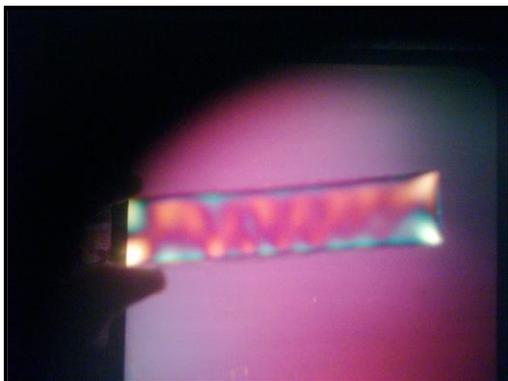


Fig. (۳) Strain pattern of heat treated glass at ۶۵۰°C, cooled in air jet to ۲۵sec. (its ambient temperature is ۱۷°C), and quenched in oil bath.

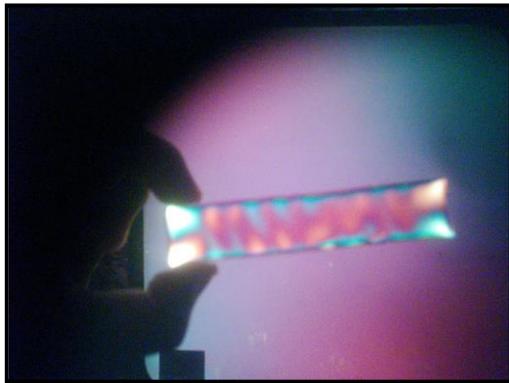


Fig. (۴) Strain pattern of heat treated glass at ۶۵۰°C, cooled in air jet to ۱.۵min. (ambient temperature is ۱۷°C), and quenched in oil bath.

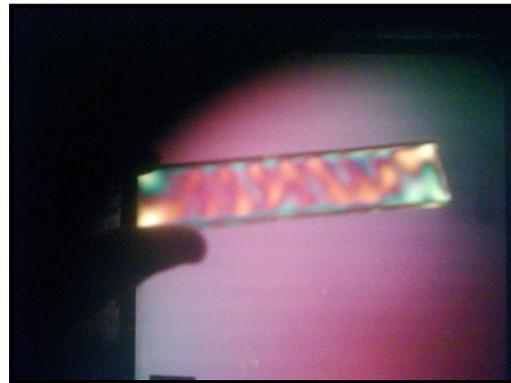


Fig. (۵) Strain pattern of heat treated glass at ۶۵۰°C, cooled in air jet to ۱min. (ambient temperature is ۱۷°C), and quenched in oil bath.

Fig.(۶) Strain pattern of heat treated glass at ۶۵۰°C, cooled in air jet to ۲min. (ambient temperature is ۱۷°C), and quenched in oil bath.

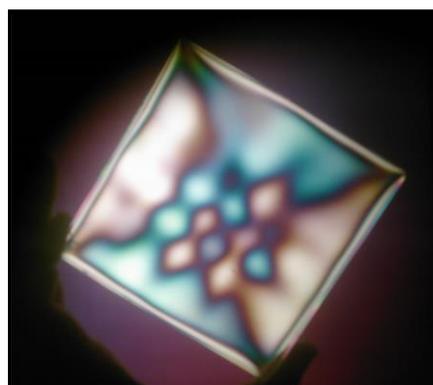


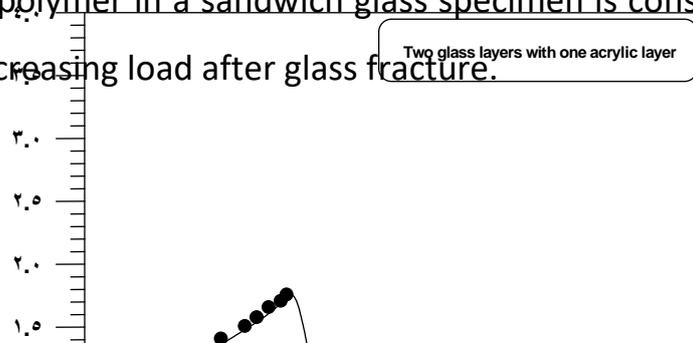
Fig. (V) Strain pattern of impact glass that heat treated at 100°C , cooled in air jet to 2°sec . (ambient temperature is 17°C), and quenched in oil bath.

0.0 Laminated Glass

laminated glass specimens are prepared and tested by Flexural test, static toughness, dynamic toughness, and shooting test. In each test the samples exhibit different behavior and following explanation of each behavior.

0.0.1 Flexural loading

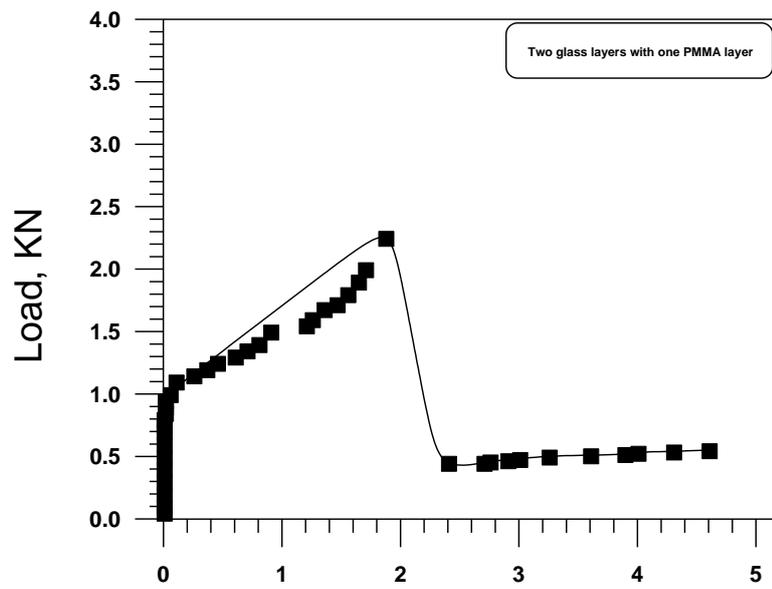
Experiments are done to achieve flexural test on laminated glass specimens. Vertical displacement has been read using a digital gauge for each reading of loading. Figures (0-17, 0-18, 0-19, and 0-20) show relationship between load and the vertical displacement for the laminated glass. Figures (0-17 and 0-18) were for two glass layers with one acrylic. Figures (0-19 and 0-20) were for three glass layers with two acrylic. The state of glass in Figures (0-17 and 0-19) is as received while in Figures (0-18 and 0-20) is treated (etched and heat treated) glass. In all Figures, vertical displacement increases with increasing applied load till maximum loading, where we have fracture of glass. The presence of polymer in a sandwich glass specimen is considered to be the reason for the increasing load after glass fracture.



Load, KN

Vertical displacement, mm

Fig. (9-17) Relationship between load and the vertical displacement of untreated glass with one acrylic layer.



Vertical displacement, mm

Fig. (٥-١٨) Relationship between load and the vertical displacement of treated (etching with heat treatment) glass with one acrylic layer.

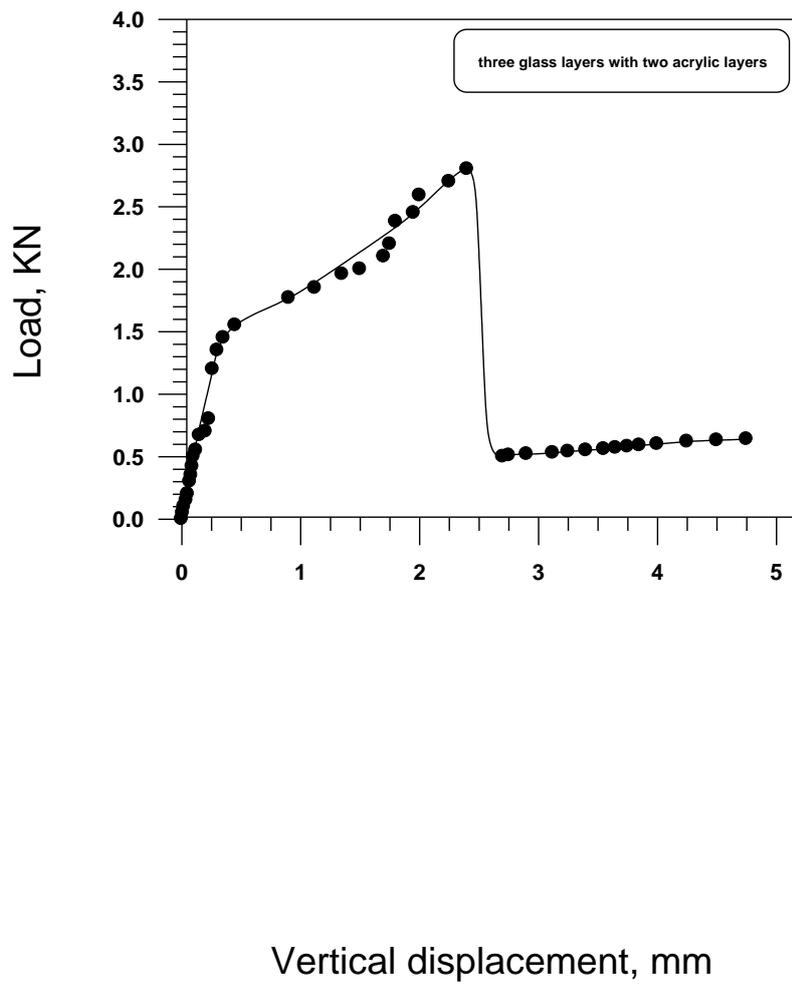
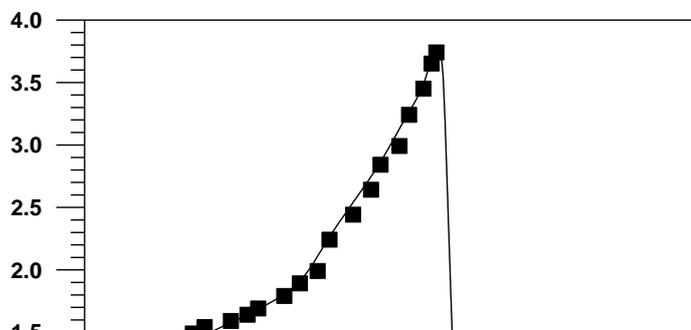


Fig. (٥-١٩) Relationship between load and the vertical displacement of untreated glass with two acrylic layers.



Load, KN

Vertical displacement, mm

Fig. (٥-٢٠) Relationship between load and the vertical displacement of treated (etching with heat treatment) glass with two acrylic layers.

Table (٥-٧) shows the different displacement of glass laminates by the different applied load. It is easy to see, that the treated glass laminates have a wider displacements and resist heavier load. This behavior is matching with the results of the researchers F. A. Veer and S. M. de Vries, in addition with Hobbelman et al [٢٤, ٢٥].

Table (٥-٧) fracture load and displacement at glass fracture of treated and untreated laminated glass.

No. of Figures	Glass state	Number of layers	Fracture load, KN	Displacement at glass fracture, mm
٥-١٧	Untreated	٣	١,٧٥	١,٧
٥-١٨	Treated	٣	٢,٢٥	١,٨٧
٥-١٩	Untreated	٥	٢,٨	٢,٤
٥-٢٠	Treated	٥	٣,٧٥	٢,٩٥

5.5.2 Toughness measurements

5.5.2.1 Static Toughness

The static toughness represents the absorbed energy during low rate loading. The value of this term can be determined by measuring the area under curve in load-displacement relationship [٦٤]. Figures (5-١٧ to 5-٢٠) are used to count the static toughness of laminated glass specimens, where the area is measured up to maximum load.

Table (5-٨) shows the static toughness of the previously mentioned laminates. It is clear, that the static toughness of treated glass laminates is higher than untreated one for different laminate numbers. Furthermore, the results show that the absorbed energy is increased as the number of glass/acrylic layers increased.

Table (5-٨) fracture load and displacement at glass fracture of treated and untreated laminated glass.

No. of Figures	Glass state	Total number of layers	Static toughness, joules
5-١٧	Untreated	٣	٢.٧٩
5-١٨	Treated	٣	٤.١٩
5-١٩	Untreated	٥	٤.٦
5-٢٠	Treated	٥	٧.٧٣

5.5.2.2 Dynamic Toughness

At high loading rate or moving loads, toughness is known by dynamic toughness. In this study, we have used many thicknesses of acrylic glass laminated systems ranging from 0mm to 4mm. In table (2-9) the values of dynamic toughness are tabulated. It can be concluded that the dynamic toughness is increased with increasing thickness of laminated glass due to the increase of the absorbed energy.

As a comparison of the results of dynamic toughness with that of static toughness, the magnitudes of dynamic toughness are higher than those of static toughness because glass is strong under high loading rates [18].

Table (2-9) The magnitudes of dynamic toughness for laminated glass.

Configurations of laminated glass	Thickness, mm	Impact energy, Joule
One layer of untreated glass	0	2
One layer of treated (etched and heat treated) glass.	0	8
Two layers of treated (etched and heat treated) glass with one acrylic layer.	13	54
Two layers of treated (etched and heat treated) glass with two acrylic layers	16	60
Three layers of treated (etched and heat treated) glass with two acrylic layers.	21	114
Three layers of treated (etched and heat treated) glass with three acrylic layers.	24	120
Four layers of treated (etched and heat treated) glass with three acrylic layers.	29	190
Four layers of treated (etched and heat treated) glass with four acrylic layers.	32	210

Five layers of treated (etched and heat treated) glass with four acrylic layers.	۳۷	۲۳۵
Five layers of treated (etched and heat treated) glass with five acrylic layers.	۴۰	۲۵۵
Six layers of treated (etched and heat treated) glass with five acrylic layers.	۴۵	>۳۰۰

۵.۵.۳ Shooting Test

This test shows the possibility of laminated glass to stop the bullet at a firing distance of ۱۰m. The results of this test are summarized in table (۵-۱۱)- ordered by the thickness of samples. All glass that is used in these samples was soda-lime glass. The type of all samples is glass-clad plastic (acrylic). As received, etched, and heat strengthened glasses in addition to acrylic and epoxy as a glue are used to fabricate the samples of this test. Table (۵-۱۰) shows the best conditions of each strengthening process with their characteristics.

Table(۵-۱۰) shows the best performance of strengthened glasses.

Etching process	Heat treatment process
HF concentration is ۱۰٪.	Initial temperature is ۶۵۰°C.
Time is ۷۵min.	Blast with air for ۳۵ seconds at ۱۷°C air temperature then quenched in oil

In the samples (۱ to ۴), the state of glass is untreated (as received glass) and their thicknesses are ranging from ۱۶mm to ۴۰mm. All these samples shows complete penetration. The thickness of sample (۵) is ۴۰mm, and it

shows partial penetration. The state of glass in this sample was treated (by etching and heat treatment) glass.

Sample (٦) has glass in state of untreated and sample (٧) has glass in a state of treated (by etching and heat treatment). The thickness of two samples is ٤٩mm. After the test, the two samples exhibit partial penetration.

Figures (٥-٢٢ to ٥-٢٥) show photos of tested samples (٤ and ٥), and their witness plate.

In sample (٨) the glass state is treated by etching only. The sample shows partial penetration. Figures (٥-٢٦ – ٥-٣١) show laminated samples and some of their witness plates after shooting test and the last Figure shows some of bullets before and after test.

For all samples after testing the glass and acrylic layers have obvious cracking as a result of bullet impact. The vicinity region around hole of the tested sample, is cracked very heavily, and appeared as a powder, opaque of comminution region. A comminuted glass is ejected during the impact. Radial cracks have propagated away from the comminuted region to the edge of each glass layer while the acrylic is cracked during impact due to the nature of brittle fracture like glass. Acrylic layers do not show the comminuted fracture but they are ejected as a small pieces away (in front of and behind) the sample. As a matter of fact, the bullet will cause a large diameter damage in the sample.

The glue (epoxy) used to bind the glass and acrylic layers has a tendency to become weak in bonding so that the glass suffered from delamination happening during impact. Here, it should be noted, that the glue used in this work is not very active where there is no better glue in the local market.

From table (2-11), one can clearly see that the resistance to bullet through the laminated glass increase with increasing target thickness. The glass state (treated or untreated) is an important condition to limit the areal density of the laminated glass. As shown in table (2-11) the areal density (which is the ratio of the weight to the area of the sample) of sample (2) is 78.0 Kg/m^2 while that of sample (1 and 3) is 93 Kg/m^2 .

So the results of testing the samples have not matched with those in papers [26, 27], because of the difference in materials used in this study. One can conclude from table (2-11), that a sample like of (no. 2) is sufficient to resist bullet. This sample has the lowest areal density and the lowest thickness of all following samples, which resist bullet impact.

Table(2-11) Shot summary

No. of target	Glass state	Configuration	No. of layers		Target thickness, mm	Areal density, Kg/m^2	Firing distance, m	Notes	Fracture mode of samples constituents
			Glass	Acrylic					
1	As received glass	Glass-clad acrylic	2	2	16	27.8	10	Complete penetration	Small pieces of glass and acrylic
2	As received glass	Glass-clad acrylic	3	3	24	44.0	10	Complete penetration	Small pieces of glass and acrylic.
3	As received glass	Glass-clad acrylic	4	4	32	61.8	10	Complete penetration	Small pieces of glass and acrylic.
4	As received glass	Glass-clad acrylic	5	5	40	78.0	10	Complete penetration	Small pieces of glass and acrylic.

٥	Treated (By etching and heat treatment)	Glass-clad acrylic	٥	٥	٤٠	٧٨,٥	١٠	partial penetration	Very small pieces of glass or powdered and small acrylic pieces
٦	As received glass	Glass-clad acrylic	٦	٦	٤٩	٩٣	١٠	Partial penetration	Small pieces of glass and acrylic
٧	Treated (by etching and heat treatment)	Glass-clad acrylic	٦	٦	٤٩	٩٣	١٠	Partial penetration	Very small pieces of glass or powdered and small acrylic pieces
٨	Treated by etching only	Glass-clad plastic	٦	٦	٤٩	٩٣	١٠	Partial penetration	Small pieces of glass and acrylic



Fig. (٥-٢١) Front view of laminated sample before shooting test.



*Untreated



*heat treated +etched

* Thickness=٤ mm

Fig. (٥-٢٢) Front view of laminated sample after shooting test.



Fig. (٥-٢٣) Front view of laminated sample after shooting test.



Trace of bullet for untreated glass

Trace of bullet for treated glass

Figure (٥-٢٤): Front view of witness plate of ٤٠ mm thick sample after test.



Figure (٥-٢٥): Back view of the same plate with behind view.



Figure (٥-٢٦): Front view of laminated sample of ٤٩mm thick after shooting test.



Figure (٥-٢٧): Front view of laminated sample of ٤٩mm thick after shooting test.



Figure (٥-٢٨): Front view of witness plate of ٤٩mm thick sample after Shooting test.

Figure (٥-٢٩): Back view of the same plate with behind view.



Fig. (٥-٣٠) Front view of laminated sample after shooting test.

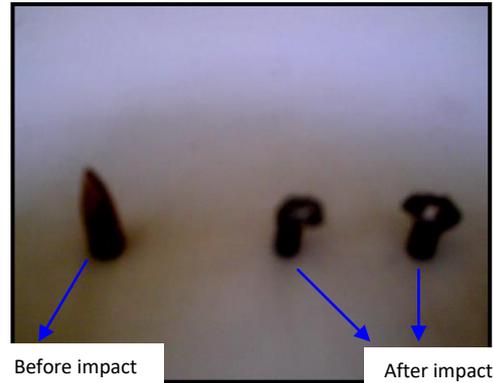


Fig. (٥-٣١) Bullets before and after impact test.

CHAPTER SIX

CONCLUSIONS AND SUGGESTIONS

٦.١ Conclusions:

On the basis of this study, measurements and observations performed, the following findings can be concluded:

١. ٤٠ mm thick laminated glass which contains treated (etched and heat treated) glass can be used to avoid a bullet of Kalashnikov rifle about distance of ١٠ m.

- ٢. Heat treatment decreases hardness of the annealed glass. ٥ percent is the decrement of hardness due to the heat treatment at the same conditions that mentioned above.
- ٣. Etching can increase the fracture strength of glass by decreasing the flaws density on the glass surface. The increment in HF concentration and etching time with constant of HF concentration can decrease the fracture strength of glass because of the over etching. In this study the best etching is at ١٠٪ HF for ٧٥min.
- ٤. Etching can raise the hardness of glass by creating a new surface with a relative smooth. ٥ percent is the increment in the hardness of etched glass at ١٠٪ HF for ٧٥min.
- ٥. The effect of etching on the heat treated glass does not sensibly increase the strength of heat treated glass but the hardness is relatively modified.
- ٦. Toughness of glass can be increased by combining it with a polymer because the polymer makes bridge among cracked glass and it holds the pieces of cracked glass together.
- ٧. The using of toughened glass in laminated glass attributes to raise static and dynamic toughness, in addition, the increasing of layers number can increase laminated glass toughness.
- ٨. The fracture strength of glass can be raised by heat treatment. The best conditions that the study is suggested are heating the glass up to ٦٥٠°C and then rapidly cooled by air jet for ٢٥ seconds (air temperature is ١٧ °C), finally quenched in oil.

٦.٢ **Suggestions:**

١. Using another type of glass such as Vycor™ ... etc.
٢. Using Ion-exchange in place of heat treatment.
٣. Mixing HF acid with other acids such as H₂SO₄ ...etc to modify glass surface smoothness.

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