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Study the Effect of particle size on the glass coating by electrophoretic deposition method

BSc thesis submitted to the Department of Ceramics Engineering and Building Materials as a fulfillment of Bachelor degree in Ceramics Engineering

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بسم الله الرحمن الرحيم

آمَنَ الرَّسُولُ بِمَا أُنْزِلَ إِلَيْهِ مِنْ رَبِّهِ وَالْمُؤْمِنُونَ كُلٌّ آمَنَ بِاللَّهِ وَمَلَائِكَتِهِ وَكُتُبِهِ وَرُسُلِهِ لَا نُفَرِّقُ بَيْنَ أَحَدٍ مِنْ رُسُلِهِ وَقَالُوا سَمِعْنَا وَأَطَعْنَا غُفْرَانَكَ رَبَّنَا وَإِلَيْكَ الْمَصِيرُ (285) لَا يُكَلِّفُ اللَّهُ نَفْسًا إِلَّا وُسْعَهَا لَهَا مَا كَسَبَتْ وَعَلَيْهَا مَا اكْتَسَبَتْ رَبَّنَا لَا تُؤَاخِذْنَا إِنْ نَسِينَا أَوْ أَخْطَأْنَا رَبَّنَا وَلَا تَحْمِلْ عَلَيْنَا إِصْرًا كَمَا حَمَلْتَهُ عَلَى الَّذِينَ مِنْ قَبْلِنَا رَبَّنَا وَلَا تُحَمِّلْنَا مَا لَا طَاقَةَ لَنَا بِهِ وَاعْفُ عَنَّا وَاغْفِرْ لَنَا وَارْحَمْنَا أَنْتَ مَوْلَانَا فَانْصُرْنَا عَلَى الْقَوْمِ الْكَافِرِينَ(286).

صدق الله العلي العظيم

(سورة البقرة)

Certification

I certify that this graduation project entitled " Study the Effect of particle size on the glass coating by electrophoretic deposition method " was prepared by " Zainab Hussein shaker" under my supervision at Babylon University / College of Material Engineering / Department of Ceramic and Building Materials Engineering , in Partial Fulfillment of the Requirements for the degree of Bachelor of Science in Materials Engineering/ Ceramic. I recommend that this project be forwarded for examination in accordance with the regulation of the University of Babylon.

Signature:

Dr. Firas Jabbar

الإهداء

في ختام هذا العمل، اود ان اقدم امتناني لكل من كان له فضل في مسيرتي و ساعدني ولو باليسير.

إلى من أُفضِّلها على نفسي و ضحَّت من أجلي ولم تدَّخر جُهدًا في سبيل إسعادي على الدَّوام (والدتي العزيزة).

نسير في دروب الحياة ويبقى من يُسيطر على أذهاننا في كل مسلك نسلكه صاحب الوجه الطيب والأفعال الحسنة فلم يبخل عليَّ طيلة حياته (والدي العزيز).

إلى أساتذتي في الكلية ممن كان لهم الدور الأكبر في مُساندتي ....

إلى اختي واخواني و أصدقائي و جميع من وقفوا بجواري وساعدوني بكل ما يملكون وفي أصعدة كثيرة.

أُهديكم بحث تخرُّجي ....

الشكر والتقدير

بعد أن أتممت هذا المشروع يسرني أن أوجه شكري لكل من نصحني أو أرشدني أو وجهني أو ساهم معي في إعداد هذا البحث بإيصالي للمراجع والمصادر المطلوبة في أي مرحلة من مراحله، وأشكر على وجه الخصوص أستاذي الفاضل الدكتور (د. فراس جبار) على مساندتي وإرشادي بالنصح والتصحيح و لاقتراح موضوع المشروع ومناقشتها العلمية ومراجعته فصول المشروع ادعوا من المولى القدير أن يمن عليه بوافر الصحة والتوفيق والمواصلة الدائمة.

Contents

Abstract

Glass coatings are of interest to moderate surface functionality of ceramic and metal materials in different applications such as in healthcare field. Electrophoretic deposition is one of the attractive methods that used in this direction. In which, charged particles in a suitable solvent are deposited on a substrate utilizing the electrophoretic forces. Borosilicate glass powder of two sizes (~ 10 and 1 µm) was employed as a model in this study to be deposited on a 316L stainless steel substrate. The electrodes of the cell had an exposed area of 1 x 1 cm^2 that horizontally fixed. The applied voltage was 30 and 60V. The solvent used was isopropanol (C_3H_8O). A solid loading of 4 wt% of the glass powder was applied in all the experiments. The yield coatings were uncontinuous at a potential of 60 V for 30 and 60 s. The results showed that good tendency of coating is with lowering the size of particle size. In addition, accurate choosing of solvent plus the dispersant are of important steps because glass is dense (~ 2.5 g/cm^3) in comparison to other ceramic materials.

Introduction

Glass is an amorphous material that has unique physical and chemical properties in comparison to the crystalline ceramics. Brittleness and high density of glass make it undesired for many applications. Therefore, glass coatings on different substrates can be good option for engineering applications.

In this study borosilicate waste glass was used as a model to be deposited on metal substrate (316 L stainless steel) by electrophoretic deposition using locally-made cell of Teflon. Many parameters can influence this method of deposition. Some of them are related to the suspension (media that carries the charged glass particles) and others are related to the cell itself. Stable suspension are the key for continuous deposited film. The parameters that belong to the cell are the applied voltage, substrates, distance between the electrodes, configuration of the electrodes and the deposition time.

The features of particles employed in this method are important as well. Density, particle size and surface features of particles are of interest. Two powders of different particle sizes were involved in this work e.g. 10 and 1 µm. This characteristic will determine the solid loading needed for the suspension and coherence of the deposited film. The applied voltage, on the other hand, plays role to pull the charged towards the cathodes.

Aims of the Study

The goal of this study can be summarized into the following points:

1. Preparation of suitable suspension for the EPD method

2. Making film of borosilicate glass on a stainless steel substrate

Chapter One

Theoretical Part and Literature Review

1.1 The electrophoretic deposition (EPD)

Electrophoretic deposition (EPD) is one of the most outstanding coating techniques due to its wide potential in ceramics coating and processing technologies [6]. It can be defined as a method where charged particles move under the influence of a direct electric field towards an oppositely charged electrode and coagulate there to form a stable deposit. Among the ceramic coatings applied by EPD are e.g., phosphors coatings for SOFCs, photovoltaic applications or insulating layers [7].

The EPD technique can be applied to any solid available in the form of a fine powder (<30 μm) or a colloidal suspension. It may carry out using aqueous and non-aqueous solvents or may be susceptible to electrolysis, usually at low electric fields. However, this method need more work needs to develop a full, quantitative understanding of the fundamental mechanisms of EPD to optimize the working parameters for a broader use of EPD in materials processing. A fundamental requirement for a successful application of the EPD technique is the preparation a stable suspension of particles in a suitable solvent [7]. The main advantages of this technique are:

- Its high versatility when used with different materials and their combinations;
- Its cost effectiveness, because it requires simple and cheap equipment.
- It can be used to coat objects with a complex shape and on small scale.

EPD is a two-step process. In the first step, charged particles suspended in a liquid medium move towards the oppositely charged electrode under the effect of an externally applied electric field (electrophoresis). In the second step, the particles deposit on the electrode forming a more or less thick film, depending on the process conditions (concentration of particles in solution, applied electric field, time). The substrate acts as an electrode and the deposit of particles is the coating [6].

1.2 Process Variables

Two sets of parameters can affect both the method and the resulting specimens as listed in table 2.1.

- Parameters that control the status and quality of suspension.
- Parameters that qualify the whole process like the applied electric field.

Table 2.1: EPD parameters

Suspension parameters	Process parameters
Particle size Concentration of solid Dielectric constant Viscosity Zeta potential	Applied electric field Deposition time Conductivity of the substrate Distance between the substrate

Hereafter is explanation of the EPD parameters.

1.2.1 Parameters related to the suspension

- **Particle size**

  The particle size has a big influence on the EPD. In case of larger particles, there is always a competition between the gravity and electrophoresis forces. The first one result in sedimentation of the powder while the latter deposits the particles on the substrate. In this situation, when EPD is completed, surface charge is highly increased and/or the electrical double layer region is thickened. [21] The appropriate grain size for electrophoretic of different ceramic and clay systems in the range (1-20 μm) was determined.

- **Concentration of solid**

  Concentration of Solids in Suspension High concentrations of solids in suspension enable uniform and even deposition rates, while low concentrations lead to deposition rates proportional to electrophoretic mobility of each particle.

- **Dielectric constant and viscosity**

  Dielectric constant of the liquids must be in the range of 12–25 for deposition to happen. In liquids with dielectric constants less than 12, lack of dissociative power stalls the deposition. While in liquids with dielectric constants higher than 25, electrophoresis is halted by the reduction in size of the double layer region. Generally, an ideal suspension has high dielectric constant, low conductivity and low viscosity. Table 2.2 shows viscosity and relative dielectric constant of some popular solvents, ordered from best to worst.

Table 2.2: Viscosity and DC of some EPD solvent

Solvent	Viscosity	Relative dielectric constant
Acetone	0.3087	20.7
Methanol	0.557	32.63
Ethanol	1.0885	24.55
Isopropanol	2.0439	19.92

Methanol and acetone are the first two best solvents. However, comparing these two, methanol has higher dielectric constant while acetone has a viscosity of almost half of methanol. Therefore, acetone can be considered as the best solvent for making an ideal suspension.

❖ **Zeta Potential**

Zeta potential is a physical property which is exhibited by any particle in suspension, macromolecule or material surface. It can be used to optimize the formulations of suspensions, emulsions and protein solutions, predict interactions with surfaces, and optimize the formation of films and coatings . It governs several key parameters in EPD such as the density of the deposit, particle direction and speed, and the repulsive interactions between the particles which determine the stability of the suspension. Generally, a high surface charge is needed not only to avoid particle agglomeration which increases the suspension stability and is expected to form dense and strongly adhering deposits. Most preferred is stable slurry in the bulk and unstable at the vicinity of the electrode to perform suitably EPD [11].

1.2.2 Process parameters

- **The applied electric field**

  The electric field in the method is the driving force which is partially carried by the free ions. Ideally, the applied electric field must be totally spent on advancement of the electrophoresis in a stable-current manner. Because the deposition increases in direct relation with raising the applied potential. Contradictory, too low fields are not capable of triggering the electrophoresis. While too high applied electrical fields represent sacrificing the quality of the deposits. It has been suggested that the best quality of deposits are gained at moderate applied fields [12].

- **Effect of deposition time**

  In EPD, the deposition rate starts with a linear relationship to time and then it lowers as time goes on, until the deposit is thick enough to interrupt the conductance and the deposition rate reaches plateau at high deposition times.

- **Conductivity of substrate**

  In EPD, the quality of the deposited film is strongly dependent on the conductivity of the substrate. Low conductivity of the substrate leads to both slow deposition and nonuniformity of the deposit.

1.3 Mechanisms of EPD

According to the literature, EPD mechanism comprises two-step process. In which particles first migrate to the substrate due to the applied electric field and then after complex electrochemical reactions and aggregation the deposited layer is formed (see Figure 2.1). Electrophoretic motion of the particles is stopped by the substrate and the density of the particles (deposits) will keep increasing there, due to accumulation.

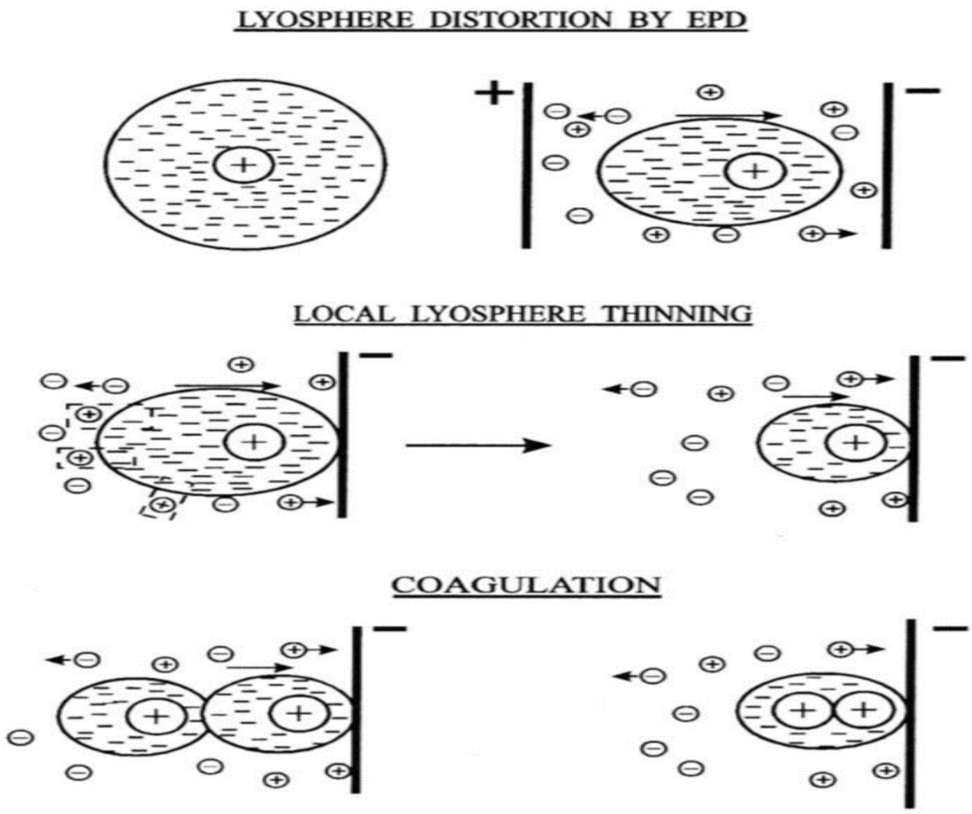

Figure 2.1: sketch illustrates the mechanism of EPD

In this mechanism, there is no guarantee that the thickness of the layer remains uniform all over the substrate. Because the nonuniformity of the electric field can lead to a very uneven deposits. If the applied electric field is powerful enough, it is also possible that deposition takes place via suppressing the electrostatic repulsion with the electric field. In this mechanism, the electrical force gradient has to be low in order to make an even deposit although some irregularities may occur due to strong electroconvection.

The next approach is flocculation of the particles by electrosedimentation. It was found that a stable suspension can produce strongly adhering sediment. Sediment is formed in the innermost layer of the particles due to gravitation plus the pressure made by electrically induced flow of particles to the substrate.

Although the density of the deposited layer is very good, the charge of the layer confronts the electrochemical charge of the solvent and acts as the driving force reducing the electrophoretic force in the layer. This instability can cause convection within the layer of particles before they are deposited on the substrate

Decrease in total ionic concentration around the substrate can raise the voltage gradient and, by quelling the convection, conduction occurs between unbalanced ions as a consequence. This effect results in raising the voltage gradient to mega volts per meter. These layers are very convectively unstable. Preservation of the surface charge of the particles results in a very strong compacting force that can make a powerful leveling effect when it is accompanied by high voltage gradient. This mechanism leads to forming very even thin layer. On the contrary, it has also been reported that an increase in ionic concentration at the substrate makes the electrostatic boundary layer of particles thinner and thinner until they become unstable and the deposit is formed The particles may lose their charge and neutralize after contacting the substrate and make a thin deposit.

This mechanism continues until the deposited layer gets thick, the particle–substrate interactions are stopped, or the pH level changes around the substrate. It has been reported that repulsive forces between the particles can be reduced only when the electrode reactions generate (OH) ions. The calculated amount of ionic strength around the substrate was sufficient for flocculation of the suspension.

Polymers is called "squeezing out" where a constant stable electrophoretic force brings two polymers into contact and deposit. Furthermore, in some cases, changing the solvent ionic composition leads to an alteration in particle surface potential and polymer particles can be stabilized Electrical double layer distortion and thinning mechanism for electrophoretic deposition [8].

EPD method are applied mostly on producing thin and thick films of different ceramics and glasses for optical, energy and medical applications.

1.4 Borosilicate Glass

Borosilicate glass was used in this study as a model for fabrication glass coatings. In fact, it has many positive properties that makes it favorable for this study. It is an amorphous solid liquids cooled at such a rate that crystals have not been able to be formed. The thermal characteristics of this glass, like low thermal expansion coefficient besides high chemical resistance against acids, drive us to design deposits on stainless steel substrates.

1.4.1 Composition of borosilicate glass (BG)

Borosilicate glass is produced using 70% - 80% Silica (SiO 2) and 7% - 13% Boric oxide (B_2O_3) with small amounts of the alkali Sodium Oxide (soda) (Na 2O) and Aluminum Oxide (AI2O3). Glassware is often used in laboratories where repeated exposure to water vapour at high temperatures can leach out alkali ions. Borosilicate glass has a relatively low alkali content and with a resultant high resistance to attack by water. Borosilicate glass has exceptional resistance to thermal shock because it has a low coefficient of expansion (3.3 x 10^{-6} K^{-1}) and a high softening point. The maximum recommended working temperature (short time) for Borosilicate glass is 500oC Borosilicate glass has good optical properties with the ability to transmit light through the visible range of the spectrum and in the near ultra-violet range. It is therefore widely used in the field of photochemistry. Because of its thermal and optical properties it is widely used for high intensity lighting applications.

This glass is used in the manufacture of glass fibres for used in plastic and textile reinforcement-- see below In the home Borosilicate glass is familiar in the form of oven-ware and other heat-resisting domestic receptacles e.g Pyrex. These items are generally used at temperatures up to 250oC Borosilicate glass has a very high resistance to attack from water, acids, salt solutions, halogens and organic solvents. It also has a moderate resistance to alkaline solutions. Only hydrofluoric acid, hot concentrated phosphoric acid and strong alkaline solutions cause appreciable corrosion of the glass. This glass is therefore widely used in chemical plants and for laboratory apparatus [2].

1.4.2 General Properties of BG

❖ **Mechanical Strength**

Glass has great inherent strength. It is weakened only by surface imperfections, which give everyday glass its fragile reputation. Special surface treatment can minimize the effect of surface flaws. The practical tensile strength of glass is about 27MPa to 62 MPa. However, glass can withstand extremely high compressive stresses. Therefore, most glass breakage is due to tensile strength failure. The reason that glass is weak in tensile strength is that it is normally covered in microscopic cracks which generate local stress concentrations.

Glass does not possess mechanisms for reducing the resulting high localised stresses and so it is subject to rapid brittle fracture. There are two methods of reducing /eliminating this problem: Treating the glass thermally or chemically such that the outer surfaces are compressively stressed at relatively high levels, the middle region between the surfaces being under tensile stress. The cracks are therefore "held closed by the continuous residual stress...This is tempered/ toughened glass. The strength of the glass can be improved by a factor of up to 10 using this method.

Ensuring that the glass surfaces have no cracks and ensuring that the glass in use is not in mechanical contact with anything which could scratch the surface. Glass produced with no surface flaws have strength values approaching the theoretical tensile strength values of 6,5 GPa. These have been produced using very fine fibres of glass. Hardness Borosilicate glass is about 2,3 x the hardness of plate glass. On the Moh's scale plate glass has a hardness value of about 5,7. Glass is harder than most grades of unhardened steel. Elasticity Gives under stress - up to a breaking point - but rebounds exactly to its original shape. Glass has virtually zero ductility. Youngs Modulus for fused Quartz glass is about 72 GPa [2]

- **Chemical resistance**

  Affected by few chemicals. Resists most industrial and food acids.

- **Thermal Shock resistance**

  Normal glass has low heat shock resistance but borosilicate glass has very good heat shock resistance, and withstands intense heat or cold as well as sudden temperature changes.

1.4.3 Recycling of Glass

Melt-quenching technique is very popular, simple and very easy to pursue the preparation of different glasses, particularly in the laboratory scale. However, this method needs high temperatures of melting which means consuming high energy even on the lab scale. Recycling of glass would be much desired on many aspects. Of these, lowering the consumption of energy and resources in addition to lowering CO_2 emission which makes industry more ecofriendly.

Industrial processes, manufacturing, and human activities are always accompanied by hazardous and non-hazardous waste materials. Thus, recycling and reprocess of such wastes is essential for environmental protection and maximizing economic benefits. Recycling is the selection, classification, and reemployment of waste as a raw material to produce the same, or very similar product, to the parent material. For instance, Mud waste from zinc hydrometallurgy [22], glass waste from lamp, bottles, and other glass products have been used in a production of glass-ceramic composite [17]. Glass waste is abundant particularly in urban areas due to intensive usage of glass in many daily life activities. Borosilicate glass, in particular, has a wide range of applications due to its chemical durability and low thermal expansion, that vary between domestic purposes (Pyrex cooking wares), scientific purposes (laboratory glassware), medical purposes (glass ampoule), and industrial applications involving reshaping of borosilicate glass tubes [18].

Such excessive usage of borosilicate glass produces a large amount of waste [19]. Moreover, glass waste should get good attention for environmental protection, especially it takes millions for year for glass to degrade in nature. So, the recycling of borosilicate glass represents a great issue that should be studied and could have great economic and environment impacts. there is no specialized industry that target the collection and recycling the waste glass. Glass waste can be utilized as fluxing material instead of common fluxes such as feldspar to reduce energy in glass ceramic production [20].

Reduction of energy consumption is an add-up advantage for using glass in the ceramic industry which will lead to reducing the overall cost of ceramic materials.

1.5 Literature Review

Ahmadi M. and Aghajani H. [2017] have reported on fabrication of Yttria-Stabilized Zirconia (YSZ) which is the most common material for thermal barrier coatings. Suspensions of 3 mol % YSZ nanoparticles in acetone medium have been prepared in presence of different amounts of iodine as dispersant. Size distribution of particles in the suspensions and zeta potential were measured as a function of dispersant concentration. Adding 1.2 g/l iodine was found to be effective for the dispersion of YSZ nanoparticles in acetone. The stability of YSZ suspension in acetone increased with iodine content increasing until reached 1.2g/l. Mean diameter of particles and zeta potential of the YSZ suspension in acetone were 912 nm and 2.4 mV respectively, and with addition of 1.2 g/l iodine shifted to 111.6 nm and 50.2 mV respectively. Electrophoretic deposition (EPD) process has been carried out from this suspension at different applied voltages and deposition times. A uniform green coating was obtained at voltage of 6 V and deposition time of 2 min the thickness of the green coating is measured about 25 µm [12].

In another work belongs to **Ghane A.S. and Aghajani H. [2018].** They used Al_2O_3 and SiO_2 to be co-deposited on medical grade 316L stainless steel to synthesize mullite particles for protection and bio-activation of metallic substrates. Stoichiometric amount of mullite was calculated and taken based on its formula ($SiO_2.3Al_2O_3$). The prepared mixture was then milled for about an hour to reach proper homogeneity. Next, 0.3 g of mixture was mixed with 100 ml of ethanol and 0.04 g iodine was added as a dispersing agent which has the duty of generating charges on the surface of particles in suspension. Finally, the suspension was ultrasonicated for about 15 min [13].

Nanocomposite coatongs were prepared by **Horandghadim N. and Khalil-Allafi J. [2019].** In their research, natural hydroxyapatite (nHA) and nHA-20 wt.%Ta2O5 nanocomposite coatings were deposited via electrophoretic deposition (EPD) on the NiTi substrate. The suspensions of nHA and nHA-Ta2O5 nanoparticles were prepared in n-butanol as a medium with different contents (0, 4, 8, 12, 16, and 20 mL/L) of triethanolamine (TEA) as a dispersant. the EPD process was conducted by a DC power supply (PSD 2600) at a constant voltage, namely at 50, 60, and 70 V for various durations of 30, 60, and 120 s . After deposition, the desiccated specimens in the air atmosphere for 24 h (to ensure the entire evaporation of n-butanol from coating structure and surface) were sintered in a VTHK-300 vacuum annealing furnace under 1.4×10^{-6} mbar at 800 °C for 1 h [14]

For medical applications, **Nayereh Askari1 N. et at. [2020]** have deposited hydroxyapatite (HA) nanoparticles on sandblasted titanium by electrophoretic deposition method. Isopropanol-acetone suspension with 50/50 ratio was used by introducing iodine as a dispersant. The suspensions were prepared with various concentrations of iodine (0, 0.2, 0.4, 0.6, and 0.8 g/L). In the next step, the optimum content of iodine was determined to provide a sustainable suspension. Then, HA coating was deposited on sandblasted titanium surface. The current density during electrophoretic deposition (EPD) and deposition rate in different voltages were investigated. Furthermore, the adhesion strength of the coating to the substrate was measured. The results showed that 0.6-g/L iodine prepared a stable suspension and the effect of current density and potential on the deposition weight is determined. In additional, the results show a finer and narrower particle size distribution can be observed. Also, adhesion strength of the coatings to the sandblasted titanium surface is about 11 MPa [15].

Chapter Two

Experimental Work

3.1 Starting materials

That were used for prepare layer coating by used electrophoretic deposition (EPD) were summarized in Table (4-1):

No.	Raw materials
1	Borosilicate glass
2	Isopropanol (C_3H_8O), (99.8%)
3	Substrate (stainless steel 316L)
4	Vertical sonicator
5	Polyethylene glycol (PEG) (MW: 4000 g/mol)

3.2 Preparation of suspension

Borosilicate glass powder was prepared by crushing and fine grinding of waste glass. The crushed glass was cleaned and dried before grinding. 0.25 g of PEG (as dispersant agent) was added to 125cc of isopropanol. They were mixed together on a magnetic stirrer for 1 h to dissolve EPG. Next, solid loading of 4 wt% (0.1 g/L) of the glass powder was added to the mixer. Eventually, the final mixture were sonicated for 15 min to generate what is called the suspension. The ultrasonic waves helped to break up the agglomerates and uniformly disperse the glass particles in the solvent.

3.3 Preparation of EPD cell

Two electrodes were horizontally fixed in the cell. This is to ensure that the deposited particles stack homogeneously on the lower side of the upper substrate. The exposed area of the substrates was 10 x 10 mm^2. The distance between the two electrodes was 10 mm too and kept constant for all the experiments. The cell was immersed in 125 cc of the prepared suspension.

DC current was applied to the cell using power locally-made supply model. Two voltages was used 30 and 60 V. the time of deposition was varied to 30 and 60 seconds.

3.4 Characterizations

3.4.1 Chemical composition determination

Concentration of the main components of the glass was determined using the gravity method at the geological scanning organization / Baghdad.

3.4.2 Particle size analysis (PSA)

Using the Bettersize 2000 lasers particles sizes analyzer (Bettersize instrument Ltd., Chinas at the ceramics laboratories / College of Materials Engineering/ Babylons University), the particle size distribution of glass powder was calculated.

3.4.3 Phase identification

X-ray diffraction (XRD) means was used to determine the phases of materials involved in this study. X-ray diffractometers (XRD 6000, Shimadzo, Japan, in ceramics laboratories / Colleges of Materials Engineerings/ Babylons Universities) was employed. In which Cukα radiation (λ = 1.5405 Å), with a scanning speeds of 5 deg/min from 10° to 60° of 2Θ (Bragg angle) and an applied powers of 40 kv/30 mA were set.

It is worth mentioning that other characteristics were planned to do as well. They might be but not restricted to:

1. Optical investigation by scanning electron microscopy

2. Hardness measurement

Chapter Three

Results and discussion

3.1 Characterizations of starting glass

3.1.1 Chemical analysis

The analysis showed that glass made mainly of SiO_2 with 81.3 mol% (see Table 4.1). The other constituent is B_2O_3 which considered very important in this glass. Because it gives the glass the thermal and chemical resistances.

Table 4.1: Chemical analysis of the glass powder implanted in this work

Oxides	SiO_2	B_2O_3	Al_2O_3	Na_2O
Mole ratio, (%)	81.3	9.2	4.3	5.2

Other oxides like alumina and soda appear in the Table 4.1. Due to the cost of analysis the test concerned to five oxides.

3.1.2 Particle size distribution

Figure (4.1) illustrates the particle size distribution as an accumulative curve of the waste glass. The d10, d50, and d90 of the powders are 0.308 , 1.121 and 4.725 respectively. It shows that the accumulative curve is not smooth. This can be attributed to the existence of agglomerates with different sizes although the milling conditions were carefully selected.

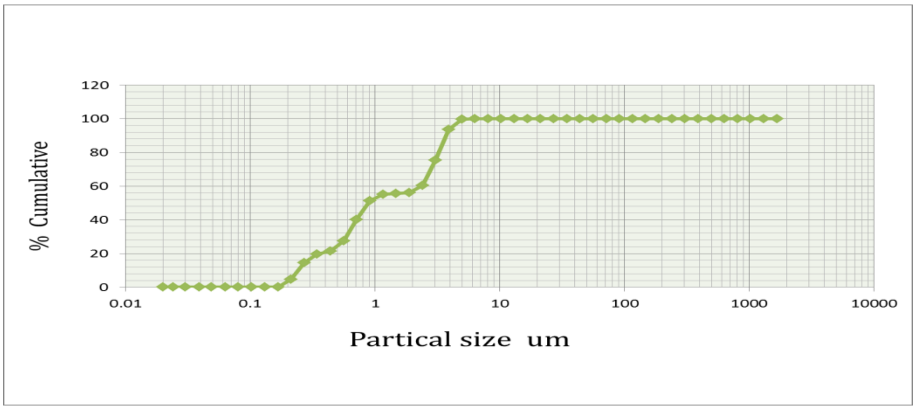

Figure 4.1: Particle size distribution of the glass powder used in this study

It was planned to do other characterizations. However, some obstacles have limit them to what stated above. Nevertheless, the author can predict some other results which be mentioned below.

3.2 Characterizations of glass film

According to the experiments I did, the thickness of the glass film nonlinearly increases with the time of deposition as represented in Figure 4.2. Where it appears rapid increase and then tends to be stable as a result of lowering the electrophoretic forces exerted on the glass particles.

Figure 4.2: Deposited glass layer

Another critical factor is the applied voltage which is solvent dielectric constant deponent.

3.2.1 Phase identification

Figure 4.3 shows x-ray diffraction of a heat treated borosilicate glass after a heat treatment at 800 °C for 1 h. Such a heat treatment was intended to do to the glass after deposition of the substrate. This treatment is necessary to increase the coherence and cohesive of the glass film on the substrate.

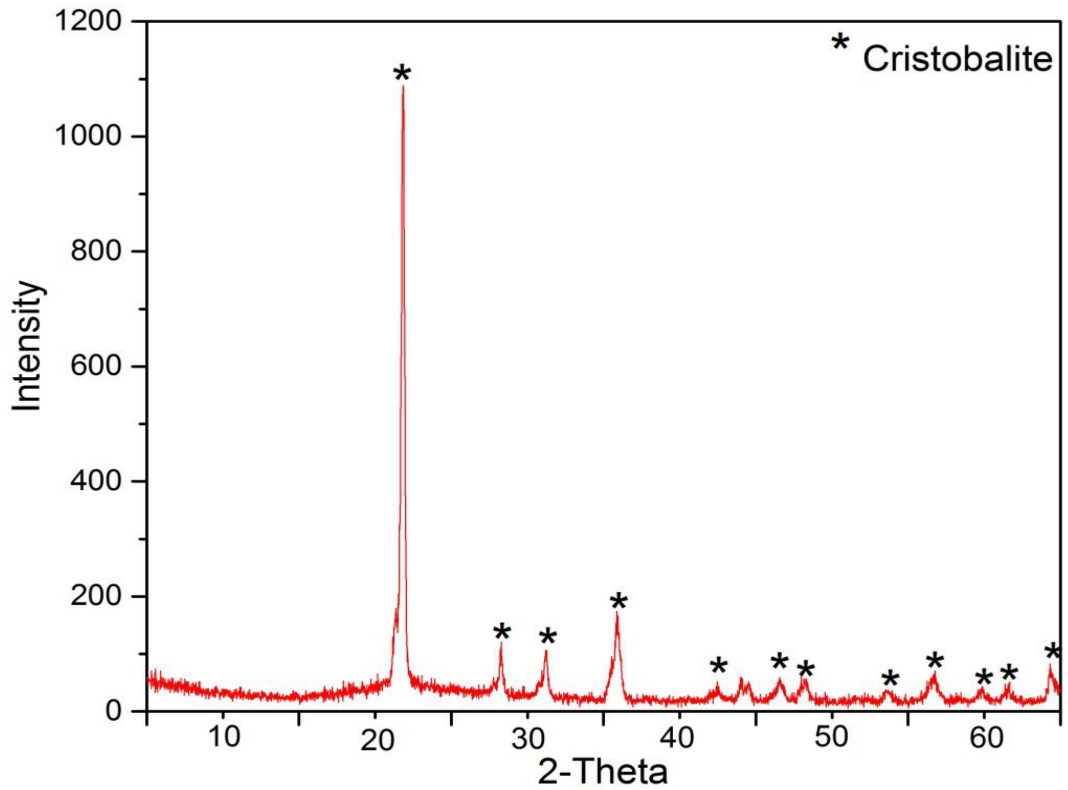

Figure 4.3: Phase identification of the heat treated borosilicate glass coating

As seen in the Figure that one crystalline phase may be formed after the annealing. The formed phase is cristobalite. The Figure appears also that there is some residual glass phase which can be seen as a hump at 2-Theta 22°.

3.2.2 Optical observation

This investigation was not done. However, inspection of the deposited film by the naked eye shows that the layers was not continuous on the substrate. The author believe that the film might appear into two cases depending on the mismatching of the thermal expansion coefficient of the substrate and the deposited glass after a heat treatment. Figure 4.4 illustrates two images of deposited glass film. The first one (Figure 4.4a) shows glass film with cracks which returns to mismatching of the thermal expansion coefficient. The other image (Figure 4.4b) is for the same film after solving the problem.

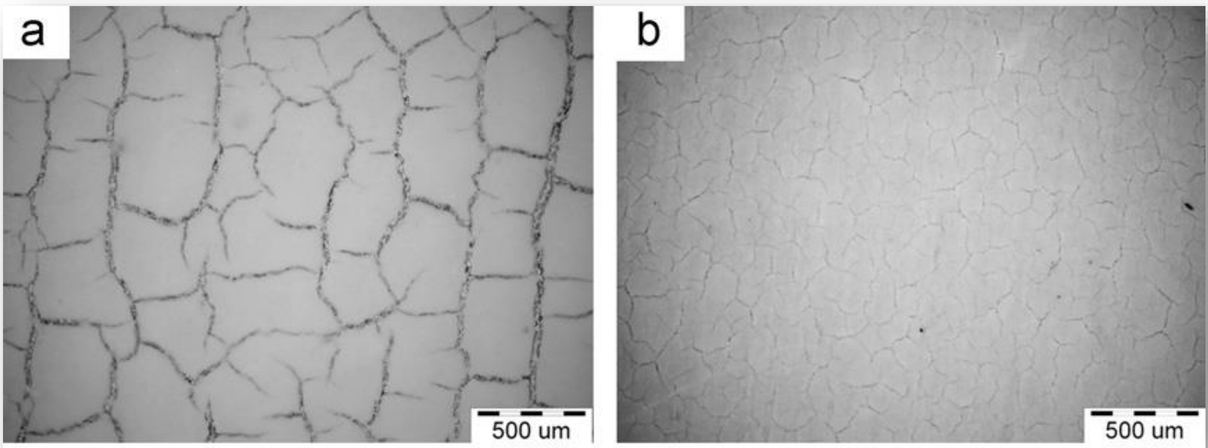

Figure 4.4: SEM images of deposited glass film by EPD method [23]

Chapter four

Conclusions and Recommendation

4.1 Conclusions

Although there is no successful glass coatings. We may conclude the following points:

1. Controlling of the method parameters leads to continuous glass coatings.
2. Preparation of stable suspension leads to continuous glass film as well.
3. Surface preparation of substrates is an important step for film adhesion.

4.2 Recommendations

1. Optimizing the parameters of the method.
2. Trying different glass composition.

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