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ABSTRACT

Title of Thesis: The Kinetics and Mechanism Studies of a
Polymer Powder Coating on Metal Surface
by Cathodic Electrodeposition Process

Wen-Lin Wu: Master of Science, May 1986

Thesis Directed by: Dr. Chen-Chong Lin

A polymer dispersion of fluorocarbon with solid content in the range of 10-25% were prepared from white powder polytetrafluorethylene (PTFE), emulsifier combination of dodecylbenzyl dimethyl sulfonium chloride and Triton N-150, and other components or additives. Such a fluorocarbon dispersion is very stable against coagulation and has a pH in the range of 8-10, which is especially suitable for powder coating purpose.

A new electrocoating technique, which is called cathodic electrodeposition powder coating method, was conducted in this study. This new coating method includes four steps: pretreatment, electrocoating, drying and baking. Two kinds of degreased metal substrates, aluminum and steel plates, were used as the cathode, while platinum was chosen as the anode in the electrocoating step. The electrocoated metal plates were subsequently dried and baked to give final transparent PTFE thin films on the metal surfaces.

Concentration coagulation would be suitable mechanism to explain the film growth of cathodic electrodeposition from PTFE dispersions. Studies of variation of film thickness with deposition time show that the film thickness at all the applied voltages used increased fastly with deposition time until the insulating characteristic started taking place to reduce the rate of film growth. Eventually, a constant ultimate film thickness was reached indicating no further deposition. Furthermore, Infrared spectrum of baked PTFE film was also measured and matched with that of PTFE melt from literature.

THE KINETICS AND MECHANISM STUDIES OF A
POLYMER POWDER COATING ON METAL SURFACE
BY CATHODIC ELECTRODEPOSITION PROCESS

By

Wen-Lin Wu

Thesis submitted to the Faculty of the Graduate School
of the
New Jersey Institute of Technology
in partial fulfillment of the requirements for the degree of
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I. INTRODUCTION

Fluorocarbon coatings are widely used in many industrial applications where non-stick, low friction surfaces are needed. They can also be used for chemical or corrosion resistance, provided conditions are not extremely harsh. These coatings can be used continually at temperature up to 288°C and intermittently to 316°C. Because of their excellent abrasion resistance and low coefficient of friction, fluorocarbon coatings are established in industrial uses, such as carburetor parts, O-rings, molds, valve components, and textile dry cans as high performance materials.

It is difficult and unsuitable to process fluorocarbon resins in molten or dissolved state. However, they are especially suited for processing in aqueous dispersions due to their fluidic behavior. The fluorocarbon dispersions are milky white liquids consisting of very small resin particles suspended in water. Generally speaking, fluorocarbon coatings can be manufactured by brushing, dipping, casting, and many powder coating methods such as fluidized bed process, electrostatic powder spraying, electrostatic fluidized bed process, and powder dusting method. Since the fluorocarbon resins are formulated in the form of aqueous dispersions, they can also be processed by electrodeposition process.

A combination of cathodic electrodeposition and powder coating, which is called the cathodic electrodeposition powder coating, was conducted in this investigation. The advantages and detailed procedures of such a new coating technique will be evaluated and described. The kinetic and mechanism of the electrocoated film growth will also be discussed in terms of transport phenomena and fusion process of particles on the metal surface.

II. THEORETICAL CONSIDERATION

A. Powder Coating

Small particles of coating material suspending in a liquid can be applied to metal surfaces using powder coating method [1]. In this method, the small discrete particles in the suspending liquid are charged electrically to one sign and the metal surface to be coated to another. Under an electric field, the particles will be accumulated on the surface of the electrode due to Coulombic attraction. Once the particles strike the surface of the electrode, they adhere and form layers of particle deposition. If the temperature of the metal is above the melting point of the powder, particles deposited on the surface will melt and adhere. If the coating process is continued until the surface temperature drops below the fusion point of the powder, the surface finish may be rough or grainy in appearance. In this case, the part may then be subjected to thermal post-treatment in an oven to further enhance flow of the applied material. With some material it may be necessary to hold the part at this elevated temperature for an additional period to allow the applied powder to develop its full characteristics.

The mechanism of film growth in powder coating process is illustrated in Figure 1 [2]. In the initial stage, the

particles approach the surface by electrical attraction. Since the plastic particles are poor electrical conductors, they will retain a portion of their charge and thus will continue to be attracted to the surface. Therefore, the particle will be held in place. In the propagation stage, subsequently arriving particles will be attracted to the surface to a greater or lesser extent depending upon the amount of charge already accumulated there due to previously deposited particles. Eventually, in the final stage, the accumulated charge on the surface will become sufficiently large so that particles will not be attracted any more. When the part has been given such a coating of powder, it is then carried into an oven where the part and coating temperature is raised to induce fusion and flow of the coating material. The part may have to be held at elevated temperature for an additional period to allow the full properties of the coating to be developed.

This method of applying powder to parts at room temperature seems to be the process which is being most widely adopted by industry at the present time. This is particularly true in those cases of furniture and appliance coating where thin films of the order of 1-3 mils in thickness are required. Where heavier films are desired, the powder is applied to the part at elevated temperature.

Three kinds of chemical materials are required to make

formulations for powder coating processes: synthetic resin powders, inorganic solid materials, and high molecular suspending agents. The aqueous dispersion latex can be readily prepared by homogeneously mixing the predetermined amounts of a synthetic resin, an inorganic solid material and an aqueous solution of suspending agent by means of a ball mill, a colloid mill, or a high speed mixer.

Both thermoplastic and thermosetting type synthetic resin powders can be used in powder coating process. Suitable synthetic resins include thermoplastic resins such as tetrafluoroethylene-ethylene copolymer, trifluoroethylene-ethylene copolymers, trifluorochloroethylene-ethylene copolymers, trifluorochloroethylene polymers, vinylidene fluoride polymers, ethylene polymers and mixtures thereof; thermosetting resins such as epoxy resins, unsaturated polyester resins and the like. Fluorocarbon resins have excellent chemical resistance to most acids and alkalies, halogen gases, oxidative chemicals, organic solvents such as halohydrocarbons, and other chemicals. They are mechanically strong in that they have high tensile strength and impact strength, excellent creep resistance and abrasion resistance. The preferred fluorocarbon resins for powder coating process are ethylene-tetrafluoroethylene copolymers having a molar ratio of tetrafluoroethylene to ethylene of 45/55 to 60/40 or ethylene-trifluorochloroethylene copolymers having a molar ratio of trifluorochloroethylene to ethylene

of 45/55 to 60/40. They preferably have particle diameters of 2-400 μm and bulk densities of 0.2-1.0 g/cm^3 .

The inorganic solid materials used in formulating aqueous dispersions include fillers or pigments, which can generally be employed for plastics, such as talc, clay, diatomaceous earth, sirasu earth, silica sand, mica, vermiculite, quartz, feldspar, gypsum, alumina, titanium oxide, graphite, carbon black, carbon fiber, and glass fiber. Generally speaking, they are added in optional quantities to adjust the physical and chemical properties of both the aqueous dispersions and their corresponding final coatings.

The most important chemicals used to prepare aqueous dispersions with good processability are high molecular suspending agents. They include those which may be generally used as a suspending agent in suspension polymerization such as cellulose derivatives, e.g., hydroxymethoxycellulose, methoxycellulose, hydroxyethoxycellulose, ethoxycellulose, sodium carboxymethylcellulose; polyvinyl alcohol, sodium .pa polyacrylate, ammonium polymethacrylate, polyvinyl methyl ether, starch, sodium alginate, gelatin, mannan, pectin, and mixtures thereof.

In those conventional methods such as powder spraying and electrostatic powder coating, only thin layers can be formed. Therefore, it is necessary to repeat the coating and

baking steps several times to manufacture a thick coating. When the coating steps are repeated, cracks are often found in the coating layer due to stresses caused by the difference in thermal expansion between the synthetic resin layer and the substrate. To overcome such a technical problem, H. Ukihashi et. al. [3] have found that the addition of 2-50% by weight of carbon fibers having a length of less than 1000 μm , uniformly mixed into the resin, allows the preparation of significantly thicker coatings in a single step and without stress cracking .

B. Electrodeposition Process

The process of electrodeposition of organic coating media to metal substrates has been known since the early thirties. The first electrodeposition process to attain commercial importance was anodic electrodeposition. In the anodic process, the metal to be coated is made the anode. The advantages of the anodic electrodeposition process are its automated character, high efficiency of coating utilization, low levels of pollution, and high throwing power, i.e., the ability to coat recessed areas in complex metal shapes such as automobile bodies. Recently, cathodic electrodeposition has assumed major commercial significance in the industrial and automotive areas. In the cathodic process, the metal to be coated is made the cathode. The

cathodic process retains the advantages of the anodic process but provides higher levels of corrosion protection.

In the manufacture of cathodic electrodeposition solutions, the synthetic resins have to be neutralized with acids and they are sufficiently soluble in water only at a pH value below 6.5 . This will somewhat lead to corrosion of the containers, tanks, pipes, pumps and other metallic components used in whole operation. The resins that are most likely used are epoxy resins with oxyalkylene structures to enhance their hydrophilic character [4,5], alkyd resins with oxazoline ring to improve the yellowing resistance [6], imino amine resins with zwitter ions to increase the film hardness [7], and acrylic polymers carrying epoxy groups [8] as electrodeposition binders. These resins can be neutralized with acids or bases. They are water soluble and can be deposited on either the cathode or the anode. On the other hand, a special group of cathodic binders is a kind of dispersion, which is stabilized with a cationic emulsifier. The dispersed molecule itself in the dispersion does not need to carry ionic groups. In the literature, acrylic dispersions containing sulfonium compounds as the cationic emulsifier are suggested [9,10].

C. Cathodic Electrodeposition Powder Coating

Cathodic electrodeposition powder coating is a new

coating technique suitable for some special water-insoluble synthetic resins which are not easy to be processed using other coating methods. In this process, the synthetic resins are formulated in the form of aqueous dispersions with cationic emulsifiers. The metal substrates to be coated are made the cathode. In order to increase the conductivity of the dispersions, auxiliary electrolytes are added, and the particles are coagulated through discharge of the stabilising emulsifier during the electrodeposition process. The deposited film after electrodeposition process is then dried and baked so that it melts or crosslinks depending on thermoplastic or thermosetting resins used. The main advantages of cathodic electrodeposition powder coating process are its high pH value of the dispersions to prevent the whole operation system from corrosion which is normally inevitable in general electrodeposition process and its higher throwing power than that in other powder coating process.

According to the literature, most of the fundamental kinetics and mechanism studies of cathodic electrodeposition were carried out on soluble polymer resin systems [11]. In contrast, information on the kinetics of cathodic electrodeposition of polymer dispersions was not available until M. S. El-Aasser et. al. first reported that of a two component system comprising mixtures of latexes of epoxy resin and amine curing agent [12]. They found that the mass

deposited increases linearly with deposition time for low applied voltages. But deposition at higher voltages occurs in two stages: an initial fast film growth stage followed by a much slower rate which represents consolidation of the deposited film.

As F. Beck [13,14] has shown, electrodeposition does not start immediately upon the application of an applied voltage or the flow of an electrical current. An induction time (τ), the order of a fraction of a second, is required to establish the necessary conditions in the boundary layer surrounding the cathode for deposition of the polymer film.

In electrolysis the amount of chemical change produced by the application of electrical current to the electrolyte is proportional to the total amount of charge passed through the cell. The chemical change produced by electrolysis may be the electrodeposition of a metal, the evolution of a gas, the oxidation or reduction of a species in solution or, in fact, any reaction that involves the transfer of a charge across an electrode interface. Assuming the deposition of polymer resin particles on a metal surface is the only main reaction involved and all other reactions can be neglected in cathodic electrodeposition powder coating process, then the amount of change is usually expressed as the weight of the deposit and Faraday's Law may be expressed by the following equation:

$$W = ItE/F \dots\dots\dots (1)$$

where W is weight of the deposit in grams,

I is current in amperes,

t is deposition time in seconds,

E is equivalent weight of the substance undergoing the electrodeposition, and

F is Faraday's constant (96,500 coulombs).

The product "It" represents the total charge passed and is expressed in coulombs.

Rearrange equation (1), by substituting $W = A \delta d$, to get

$$\delta = \frac{I}{A} \frac{E}{Fd} t = j \frac{E}{Fd} t \dots\dots\dots (2)$$

and the rate of film growth is expressed by

$$\frac{d\delta}{dt} = \frac{jE}{Fd} \dots\dots\dots (3)$$

where δ is film thickness in cm,

A is total area of the metal surface in cm^2 ,

d is density of the deposited film in g/cm^3 , and

j is current density in $ampere/cm^2$.

III. EXPERIMENTAL

A. Materials:

Fluorocarbon resin used in preparing the electrocoating dispersion was powder polytetrafluoroethylene (PTFE) purchased from E. I. du Pont de Nemours & Company, Inc.. Surfactants used in preparing the electrocoating dispersion were dodecylbenzyltrimethyl sulfonium chloride purchased from Dow Chemical Company and Triton N-150 purchased from Rohm and Haas Company. Additives used in preparing the electrocoating dispersion were Acrysol GS purchased from Rohm and Haas Company, ammonia hydroxide solution from J. T. Baker Chemical Company, and silicone antifoam agent, ASG 730, from Union Carbide Corporation. Solvents used in this experiment were trichloroethane for degreasing purpose from Polysciences Inc. and diisobutyl ketone for formulating dispersion from Eastman Kodak Company. Two kinds of metals used in this experiment as the cathode were aluminum and steel. In addition, platinum was used as the anode.

B. Instruments:

Some instruments and equipments used in this work included Infrared Spectrophotometer (Perkin-Elmer 1310), constant power supply (Buchler 3-1500), multimeter, high-

speed mixer, pH Meter, and oven with temperature control.

C. Preparation of PTFE Dispersion

Aqueous fluorocarbon dispersions of four different concentrations were prepared for this study; their compositions and weight percentages are outlined in Table 1. First, calculated quantities of dodecylbenzyltrimethyl ammonium chloride and Triton N-150 were dissolved in calculated quantity of water to give low surface tension solution (A). Second, calculated quantity of powder PTFE was mixed with calculated quantity of diisobutyl ketone to give wetted powder (B). Third, B was poured, portion by portion for two hours, into A already in a high-speed mixer with power on and mixed them for additional two hours to give stable dispersion. Finally, SGA 730 was added trace to defoam the dispersion and ammonium hydroxide solution was added to adjust the dispersion with pH = 9.0 measured by pH Meter to give the final PTFE electrocoating dispersion.

D. Cathodic Electrodeposition Powder Coating

A detailed procedure of cathodic electrodeposition powder coating is illustrated in Figure 2. There are four steps in such a new process: (1) pretreatment, (2) electrocoating, (3) drying, and (4) baking.

(1) Pretreatment:

The shape and size of the metal substrates was in the form of plane plate with 40 X 35 X 0.5 mm for aluminum and 40 X 35 X 1 mm for steel. Aluminum plates were degreased in trichloroethane for 20 minutes and then dried in air only, while the steel plates were first derusted in 15% chloric acid solution and then degreased by the same procedure as for aluminum plates.

(2) Electrocoating:

Constant power supply (Buchler 3-1500), multimeter, and electrocoating cell with PTFE dispersion were arranged as shown in Figure 2. Constant power supply was used in either constant current mode or constant voltage mode. Multimeter was used as either a voltmeter during constant current electrodeposition or a amperemeter during constant voltage electrodeposition. Three different concentrations of PTFE dispersions were poured into electrocoating cell as electrocoating bath respectively. Two equal size plane platinum plates of 40 X 35 mm were placed at each end of the cell as anodes. The metal plate to be coated was immersed equidistant between the platinum anodes as the cathode. The electrocoating was conducted under constant applied voltage condition. However, different voltages were preset and the experiment was repeatedly run with different deposition time for the two kinds of metal plates and four concentrations of PTFE dispersions.

(3) Drying:

After electrocoating process, the next step is removal of the water from the deposited film. This was done in a low temperature oven because the speed of water removal, in this case, was limited by the tendency for bubble formation. All the substrates with less than 25 μm (1 mil) coating were dried as long as 5 to 10 minutes at temperature of 75-80°C.

(4) Baking:

Drying is followed by a baking and sintering operation, during which the wetting agent is volatilized, and the powder particles are sintered into a homogeneous film. All the samples after the drying step were baked in a high temperature oven at a temperature of 380-385°C for 20-25 minutes.

E. Measurement and Testing of Baked PTFE Film

The weight of the PTFE films were obtained by weighting the metal plate before and after the coating and baking process and the thickness was then calculated assuming that the coating is uniform in thickness and its density is known to be in the range of 2.1-2.2 g/cm^3 [15]. Infrared spectrum of the electrocoated film was also measured and compared with known PTFE spectrum.

IV. RESULTS AND DISCUSSION

A. Preparation and Properties of PTFE Dispersions

The chemicals and the procedures for preparing the fluorocarbon electrocoating dispersions were described in detail in Chapter III. The fluorocarbon electrocoating dispersions are composed of very fine particles (median particle size is about 0.1-0.3 μm) of polytetrafluorocarbon in water. Cationic emulsifier, wetting agent and other additives were added to control the properties of the dispersions suitable for electrocoating process. In our study, we used a combination of both cationic emulsifier (dodecylbenzyltrimethylsulfonium chloride) and nonionic wetting agent (Triton N-150, nonylphenoxypolyethoxyethanol), which proved to be much more effective than using cationic emulsifier only. The emulsifier combination used has two principle functions: (a) to decrease the interfacial tension between the liquids, enabling easier formation of the greatly extended interface, and (b) to stabilize the dispersed phase against coalescence once it is formed. The emulsifier combination, however, has a tendency to form foam so that silicone antifoam agent (SGA 730) was added in trace amount to overcome this problem. To increase the viscosities of the dispersions for easier electrocoating, a water soluble thickener (Acrysol GS, 12% of sodium polyacrylate in H_2O)

was added for this purpose. The pH of dispersions is about 9 for electrocoating application and ammonium hydroxide solution (6M) was used to adjust the pH value required.

B. Mechanism of Cathodic Electrodeposition Powder Coating from PTFE Dispersions

The PTFE dispersions used for the present investigation were quite stabilized with adsorbed molecules of dodecylbenzyltrimethyl sulfonium chloride, which is a cationic emulsifier, fully ionized at wide range of pH. Figure 3 shows a conception of the deposition of charged PTFE particles which migrate to the metal cathode to form a porous conductive coating that is high in solids compared to the original dispersion. The porosity of the coating depends upon the polymer resin type as well as the dispersion ingredients and their interactions. This porosity, in turn, affects the resistance of the coating which controls the rate of subsequent film growth. The PTFE particles after migration to the metal cathode coagulate and adhere to its surface by a destabilization process that overcomes the small repulsive forces between the particles (concentration coagulation). A direct electrochemical reaction involving charge transfer from the particle to the electrode is not necessary for coagulation.

Figure 4 illustrates schematically the concentration gradient existing in the electrocoating process. Next to the electrode at point A is the layer already deposited, as indicated by a region of high and constant concentration. Distance B is the surface at which the actual formation of the deposit occurs and the concentration changes discretely corresponding to the transition from the deposited layer to the concentrated PTFE dispersions. The concentration decreases gradually from distance B to distance C and this represents the highly concentrated layer responsible for producing the concentration pressure required for the formation of the deposit at point B. At distance C, the concentration becomes equal to the bulk concentration of the PTFE dispersions. Initially the particles surrounding on the the surface of the electrode are gradually pressed together under the influence of the applied potential. Then layer formation of very dense structure would take place in the discrete transition zone. The smaller the critical distance between the PTFE particles, the denser would be the structure of the deposit and the greater its mechanical strength. It is the adhesiveness between the particles deposited on the plate that determines the mechanical strength and stability of the layer (i.e., only the outermost layer is redispersed on shaking).

C. Variation of Film Thickness with Time for Constant Voltage Electrodeposition Process

The electrodeposition kinetics at constant applied voltage were followed by observing current flow and extent of film growth as a function of deposition time. Typical plots of current density against deposition time at various applied voltages for PTFE dispersions on the aluminum or steel surface are presented in Figure 5. The current density for all the curves decayed towards a residual current of below 20 mA/dm². The drop in current density with time was the steepest for the highest voltage used (i.e., 4 volt). The decrease was more gradual for lower applied voltages. Thus, deposition at higher applied voltage offered rapid current cut-off as the film became insulating. The deposited weight and its corresponding thickness of the PTFE films at different applied voltages for four concentrations of PTFE dispersions on aluminum and steel surfaces are listed in Tables 2-5. Figures 6-9 show how the film thickness on aluminum surface increased with deposition time at different applied voltages in 10%, 15%, 20%, and 25% PTFE dispersions, respectively. The results given in these figures show that the film thickness increased fastly with time until the insulating characteristic of the deposited film started taking effect. At this moment, the resistance of the deposited film increased resulting in a greater potential

drop across the film which slowed down the rate of film growth. Finally, a limiting film thickness was reached indicating that there was no further deposition. This is due to the compaction of the accumulated coating film during the entire deposition period so that the conductivity of the deposited film decreased sharply. In addition, the rate of film growth at higher applied voltages was greater than that at lower voltages and this is consistent with the variation of current flow in Figure 5. The experimental data show that the rate of film growth decreased as deposition time increased. This can be explained by equation (3) in Chapter II. Since the current density dropped during the constant voltage cathodic electrodeposition process, the rate of film growth was reduced accordingly.

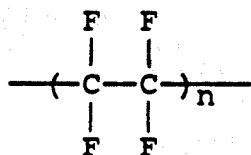
The film thickness on steel surface at different applied voltages in 10%, 15%, 20%, and 25% PTFE dispersions is plotted as a function of deposition time as shown in Figures 10-13, respectively. These plots are quite similar to those on aluminum surface and can be explained in the same way. Furthermore, the experimental fact that the limiting film thickness on aluminum surface is generally larger than that on steel surface indicates that aluminum surface can be coated more readily by electrodeposition of PTFE dispersions than steel surface.

D. Effect of Solid Content on Film Thickness

Figure 14 shows a linear relationship between the concentration of PTFE dispersion and the thickness of its corresponding film at deposition time of 2 minutes at different applied voltages on aluminum surface. It is proper to see that the higher the concentration of PTFE dispersion, the thicker was the deposited film. On the other hand, successive depositions from the same PTFE dispersion result in decreased film thickness due to the fact that the particle concentration is being depleted. PTFE dispersion particles might acquire different charge to mass ratios, and those with the highest ratio are expected to migrate and deposit faster than others. So what we might have is a fractionation based on charge to mass ratio. This depletion affects the equivalent weight of PTFE dispersion undergoing electrodeposition (E) in equation (1) and (2), thus reducing the yield of the deposit and its thickness. In electroplating, metal ions lost due to depositing on the cathode are replenished by the anode. As a result the metal ion concentration in the plating solution remains constant. However, resin is not replenished in dispersion after deposition, thus it is not surprising that cathodic electrodeposition of PTFE dispersion shows a concentration dependence.

E. Infrared Spectra of Baked PTFE Films

The Infrared spectrum of baked electrocoated PTFE film and that of PTFE melt from literature [16] are shown in Figure 15. Both of the Infrared spectra were made from dispersion PTFE melt and are quite identical each other. The chemical structure of PTFE is shown as follow:



and the characteristic absorption peaks of its main functional groups in these samples occurred as follows: C-F bending at 2400 cm^{-1} , C-F stretching in the range of $1000\text{--}1250 \text{ cm}^{-1}$. The slight difference between the two Infrared spectra is due to a trace carbonaceous residue that remained in these films during baking step. Most of the emulsifiers and additives were volatilized at the baking temperature of 380°C , but a small amount remained and decomposed during sintering, leaving a minute carbonaceous residue that could be removed by oxidation if the sintering time was extended.

V. CONCLUSIONS

1. PTFE dispersions with four different concentrations were prepared from powder polytetrafluorocarbon, which were suitable for cathodic electrodeposition powder coating to give transparent thin films on aluminum and steel surfaces.
2. The mechanism of film growth in constant voltage cathodic electrodeposition powder coating from PTFE dispersions can be explained by the concentration coagulation.
3. The rate of film growth in constant voltage cathodic electrodeposition powder coating from PTFE dispersions on metal surfaces decreased as the thickness of the film increased, resulting in ultimate film thickness which implies no more deposition.
4. PTFE thin film fabricated by cathodic electrodeposition powder coating process has a thickness up to 14 μm , which can be used for corrosion resistance applications.

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VII. GLOSSARY

- τ : Induction time [sec]
- W : Weight of the deposit [gram]
- I : Current [ampere]
- t : Deposition time [sec]
- E : Equivalent weight of the substance undergoing the electrodeposition [-]
- F : Faraday's constant [96,500 coulombs]
- δ : Film thickness [cm]
- A : Total area of the metal to be coated [cm²]
- d : Density of the deposited film [g/cm³]
- j : Current density [ampere/cm²]

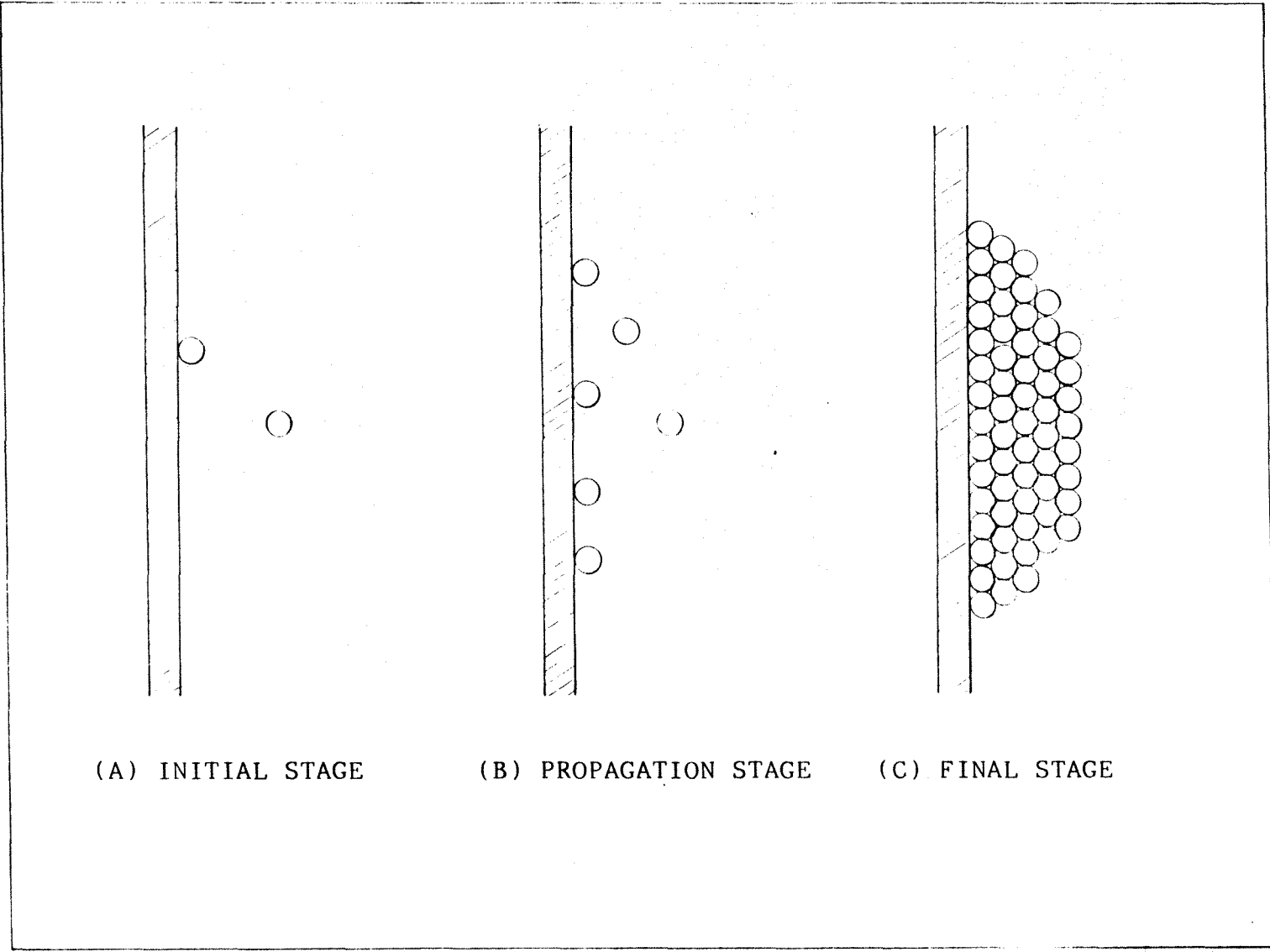


Figure 1: Mechanism of Film Growth in Powder Coating Process

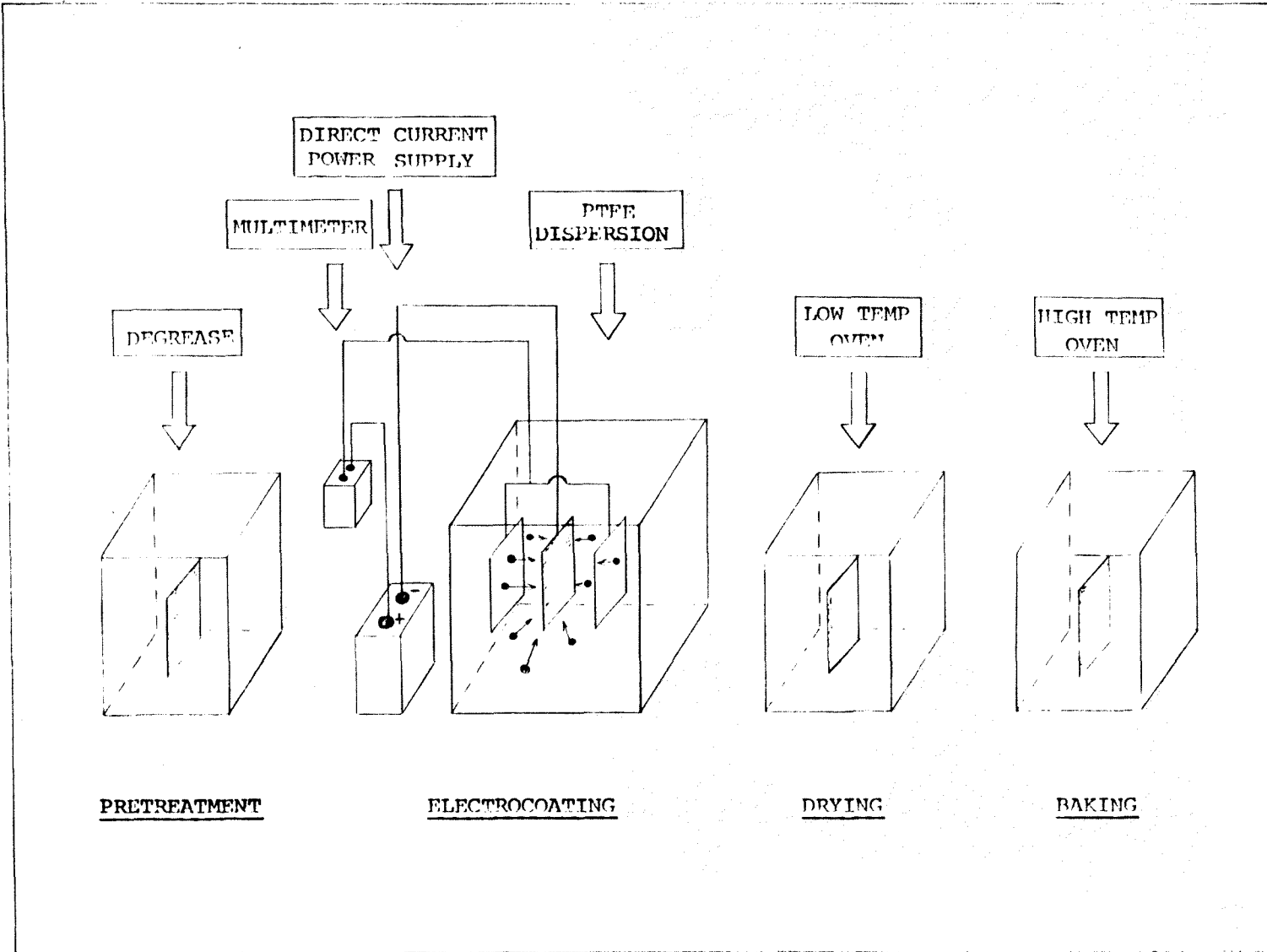


Figure 2: Experimental Setup for Cathodic Electrodeposition Powder Coating Process

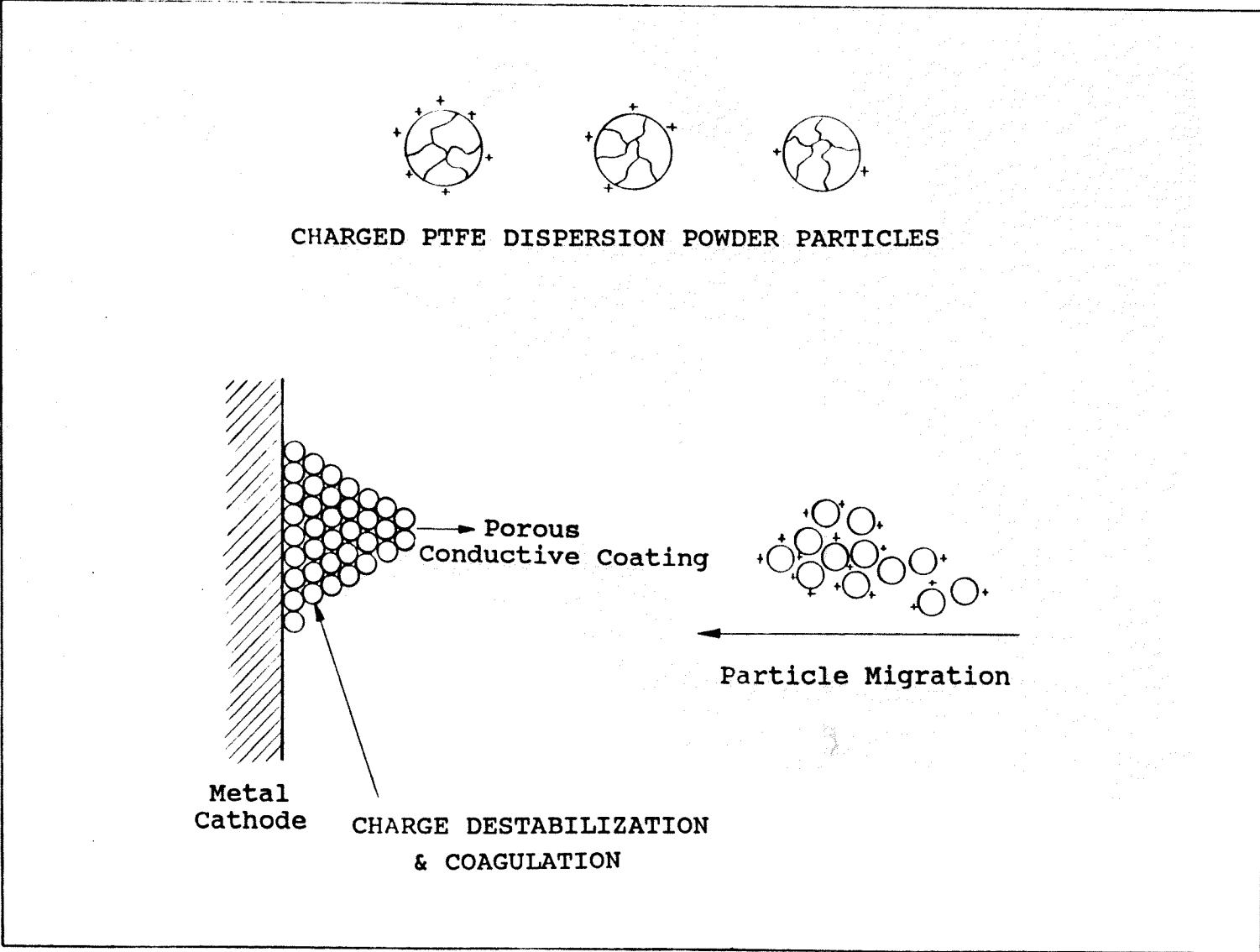


Figure 3: Schematic of Charged PTFE Dispersion Powder Particles during the Electrocoating Process

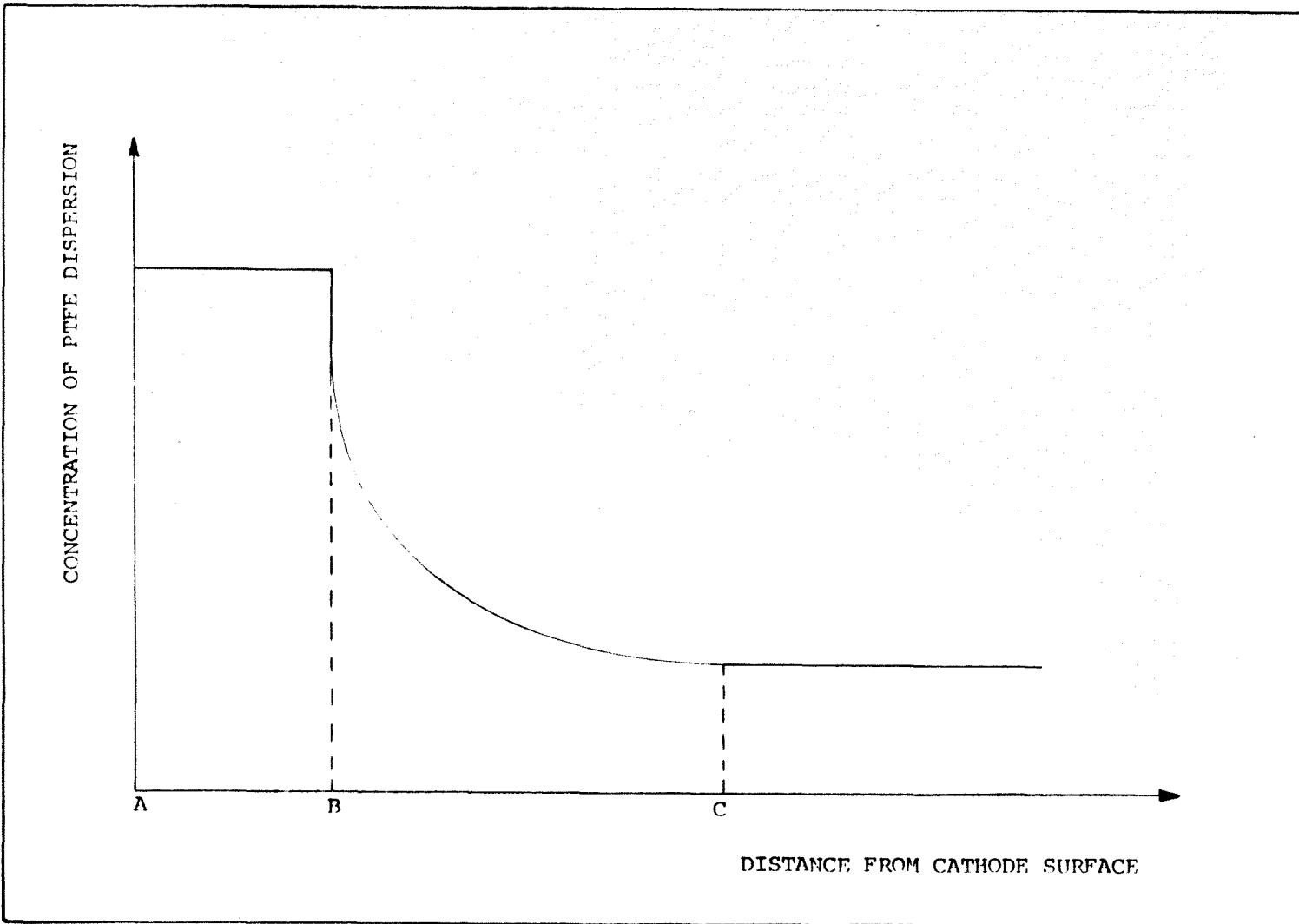


Figure 4: Concentration Profile of the PTFE Dispersion Next to the Cathode during the Electrocoating Process

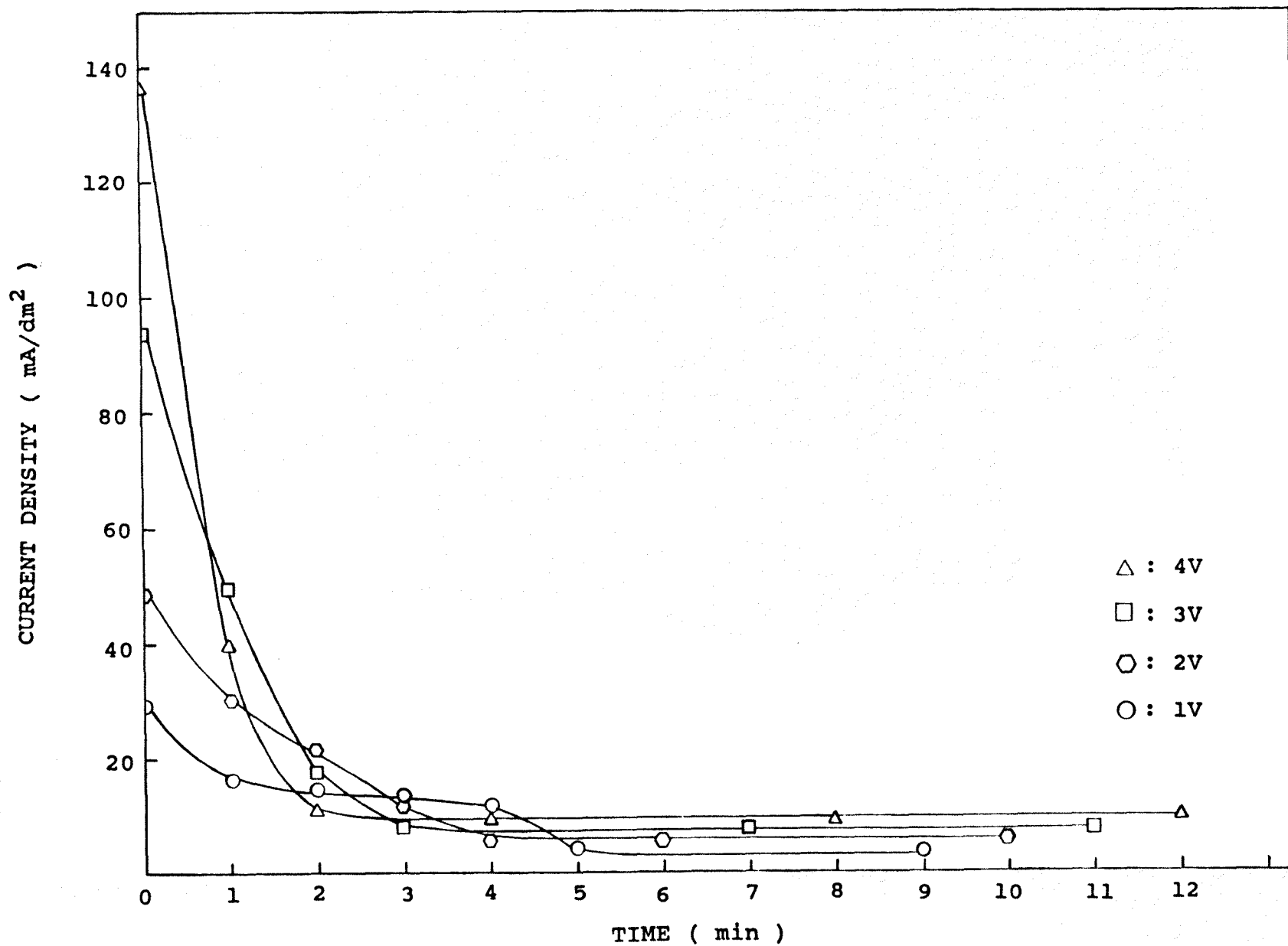


Figure 5: Variation of Current Density with Deposition Time for Various Applied Voltages

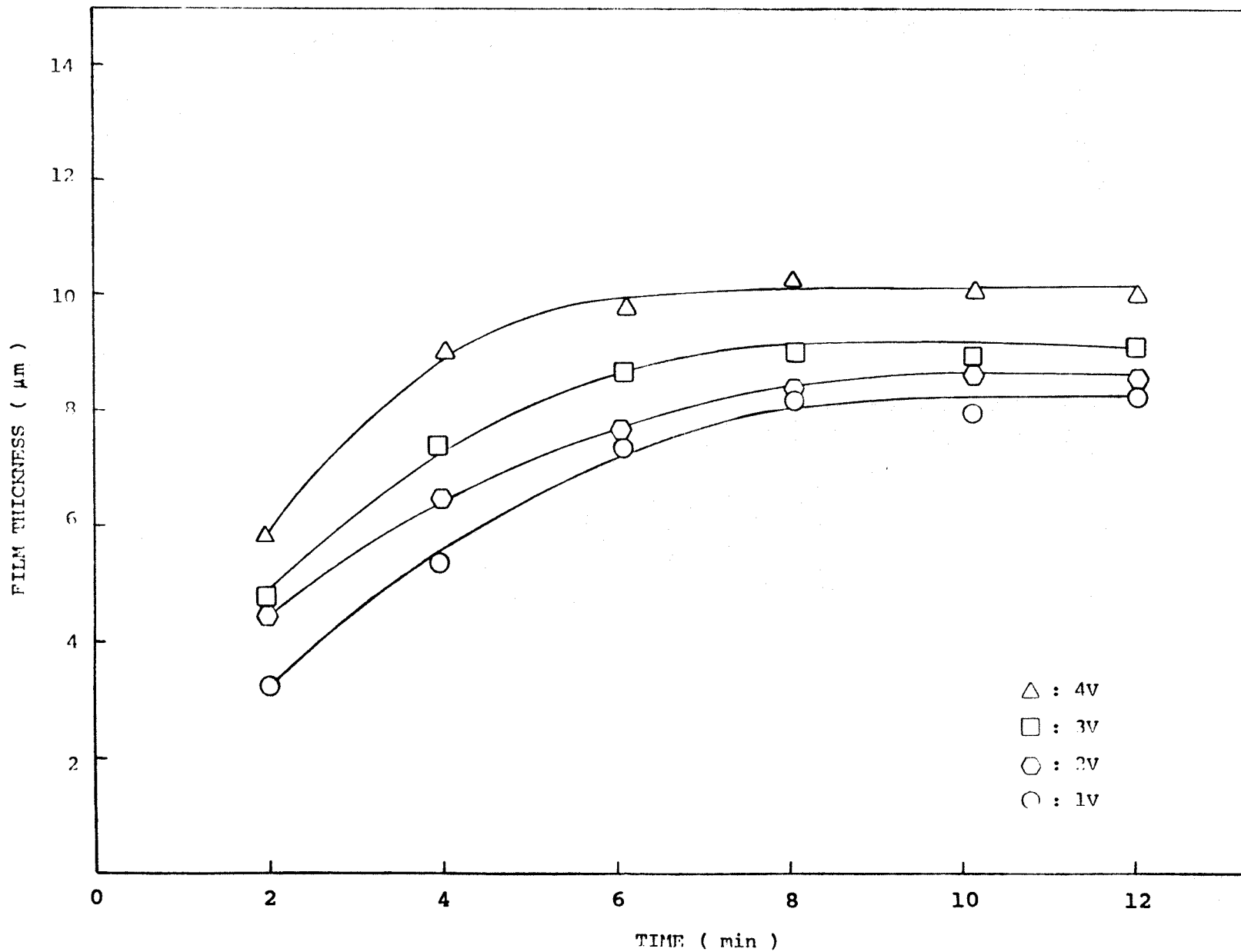


Figure 6: Variation of Film Thickness with Time for Constant Voltage Cathodic Electrodeposition using 10% PTFE Dispersion on Aluminum Surface

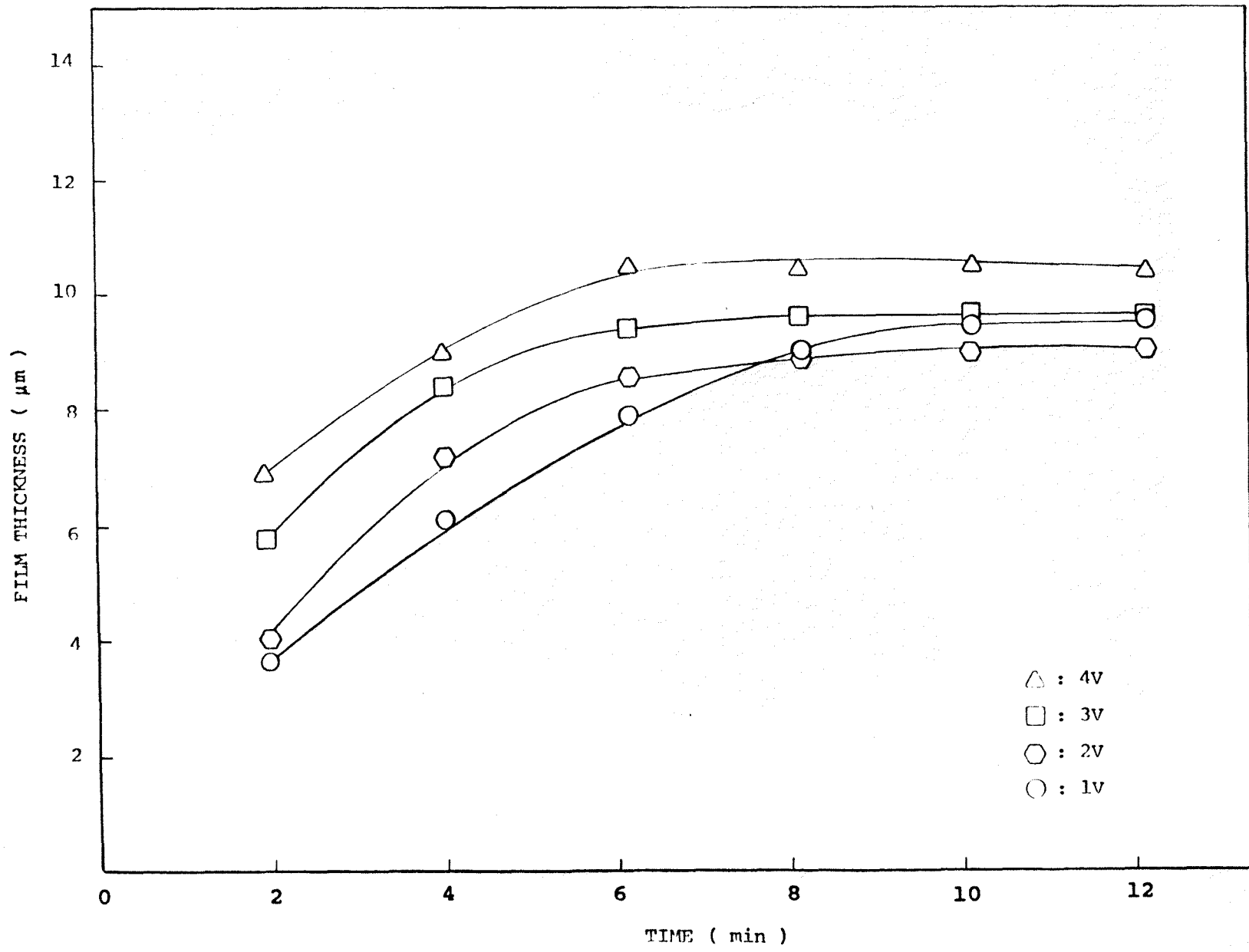


Figure 7: Variation of Film Thickness with Time for Constant Voltage Cathodic Electrodeposition using 15% PTFE Dispersion on Aluminum Surface

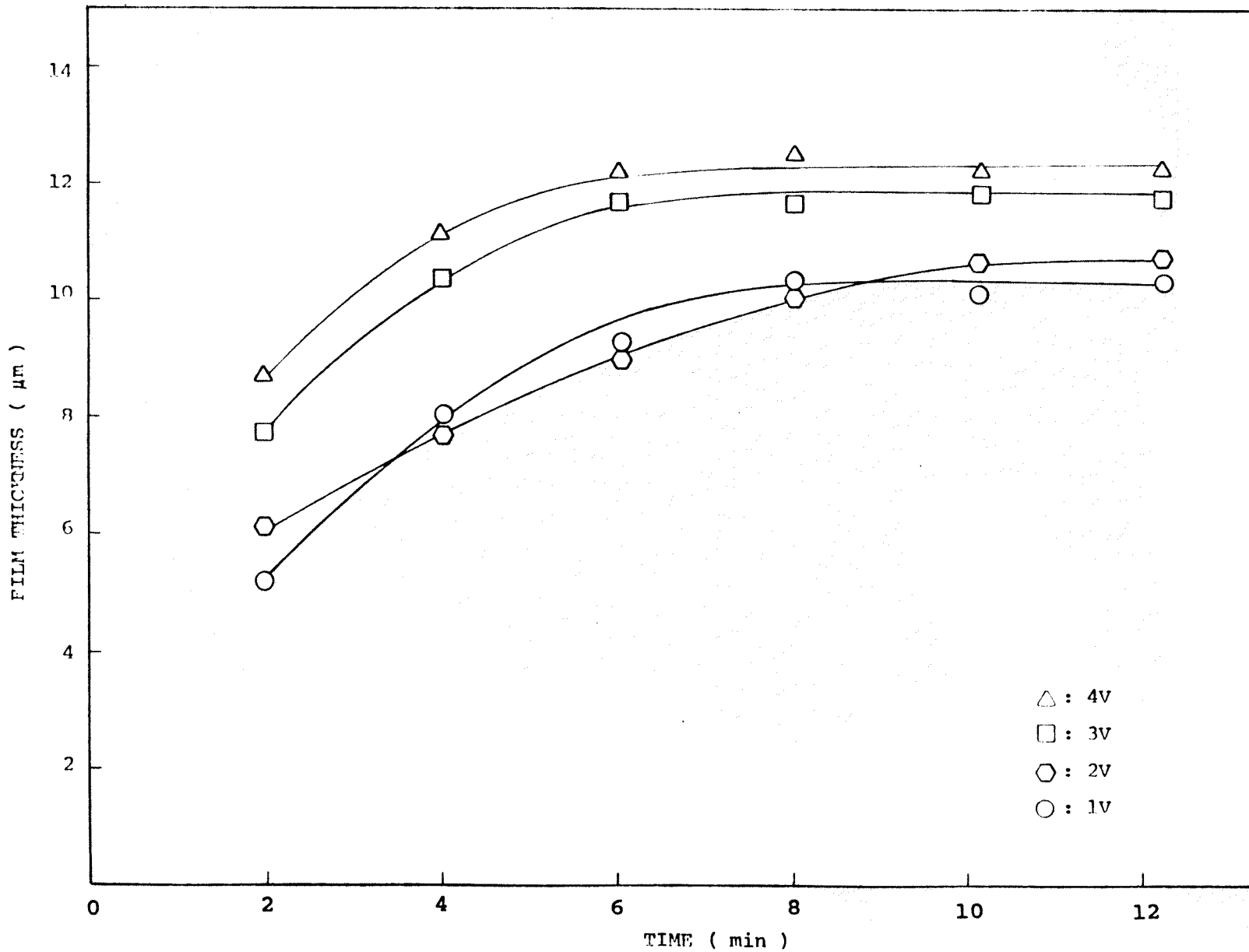


Figure 8: Variation of Film Thickness with Time for Constant Voltage Cathodic Electrodeposition using 20% PTFE Dispersion on Aluminum Surface

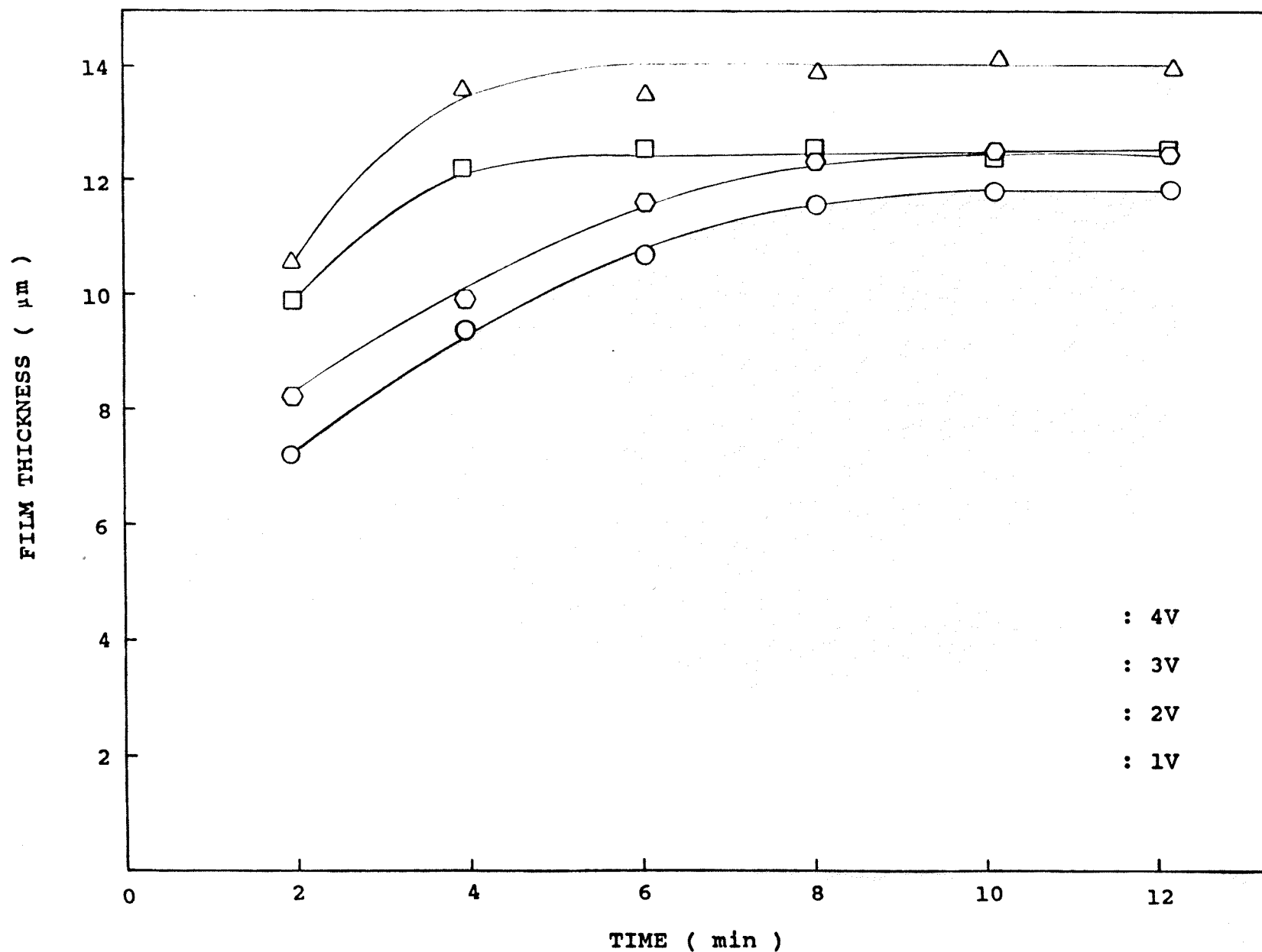


Figure 9: Variation of Film Thickness with Time for Constant Voltage Cathodic Electrodeposition using 25% PTFE Dispersion on Aluminum Surface

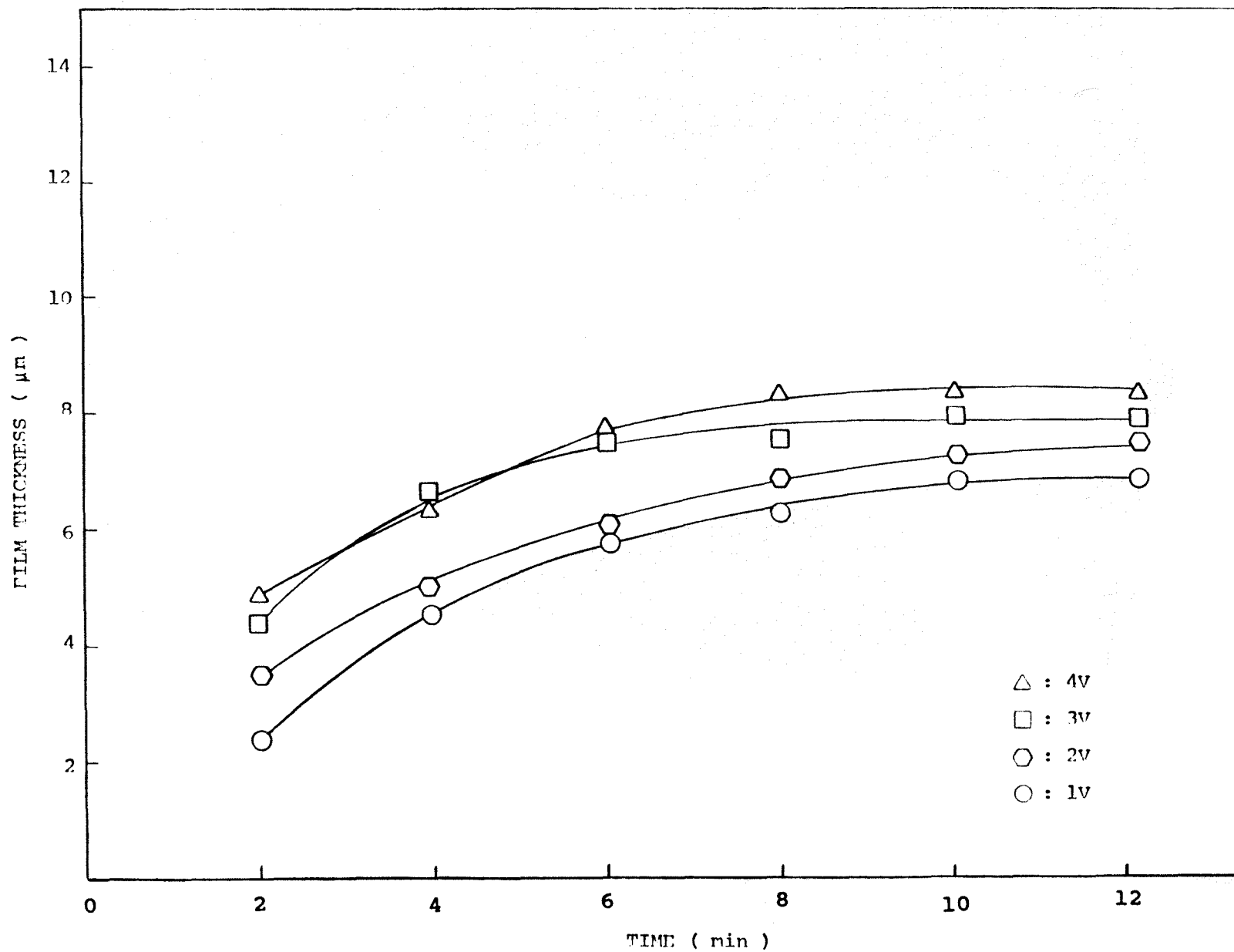


Figure 10: Variation of Film Thickness with Time for Constant Voltage Cathodic Electrodeposition using 10% PTFE Dispersion on Steel Surface

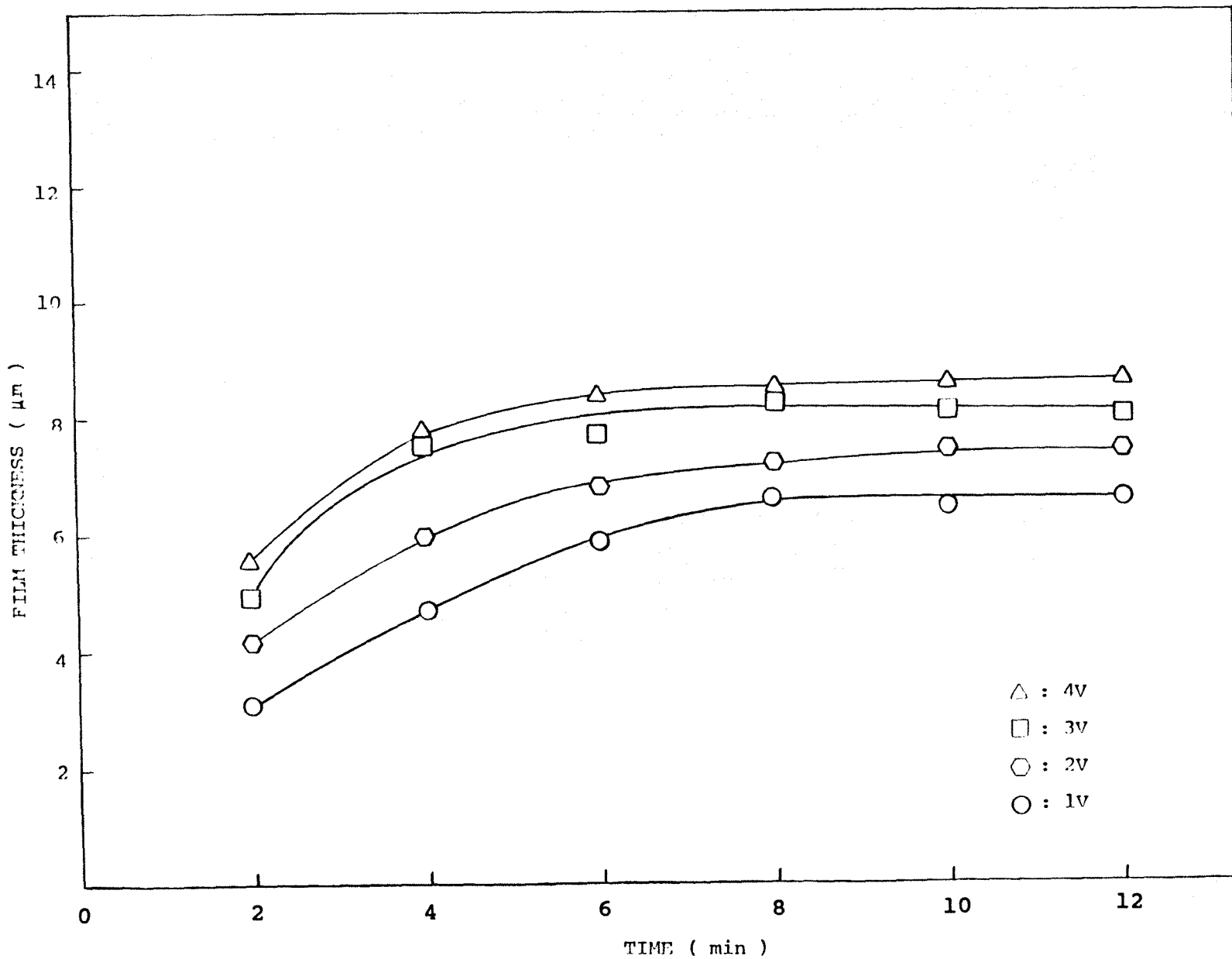


Figure 11: Variation of Film Thickness with Time for Constant Voltage Cathodic Electrodeposition using 15% PTFE Dispersion on Steel Surface

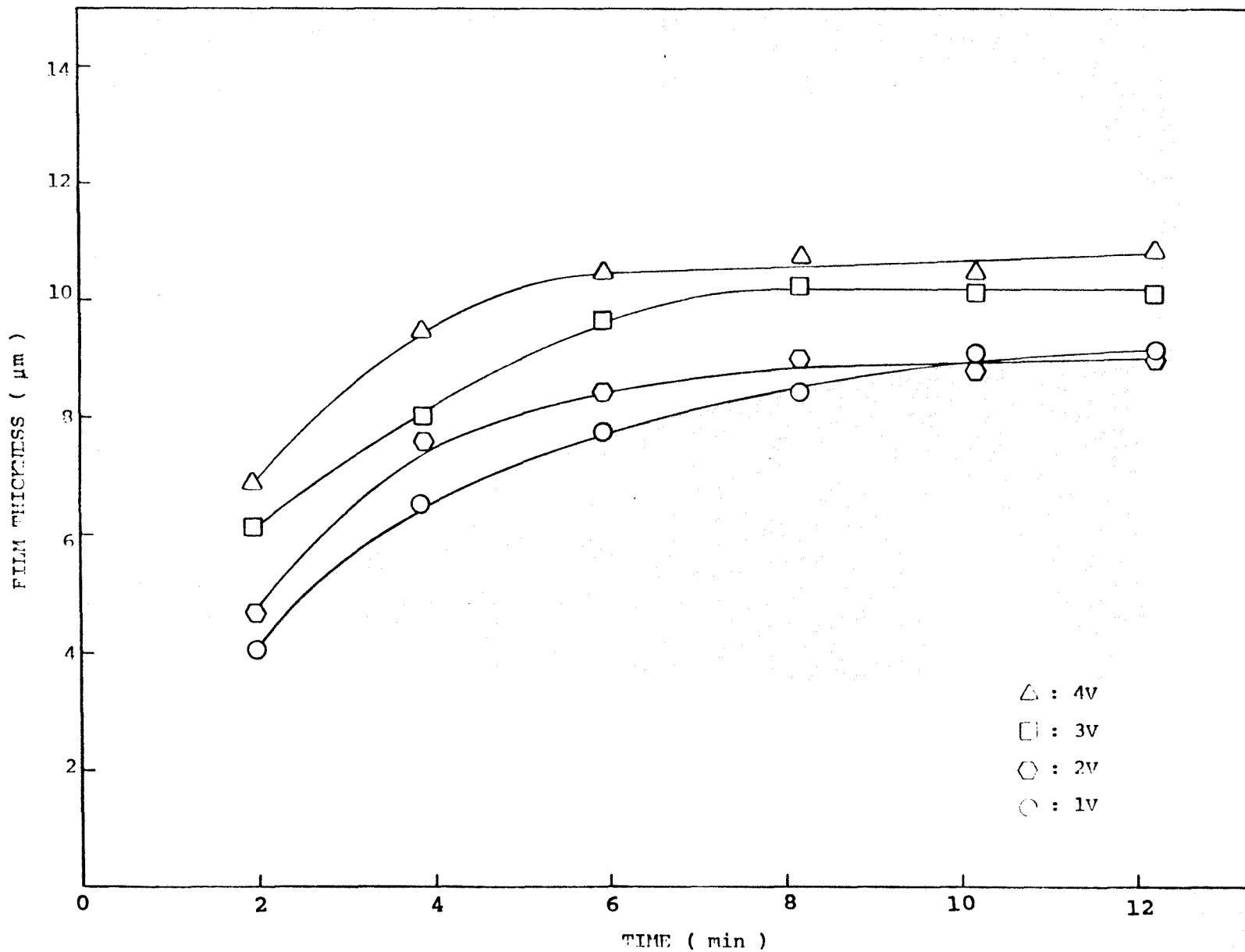


Figure 12: Variation of Film Thickness with Time for Constant Voltage Cathodic Electrodeposition using 20% PTFE Dispersion on Steel Surface

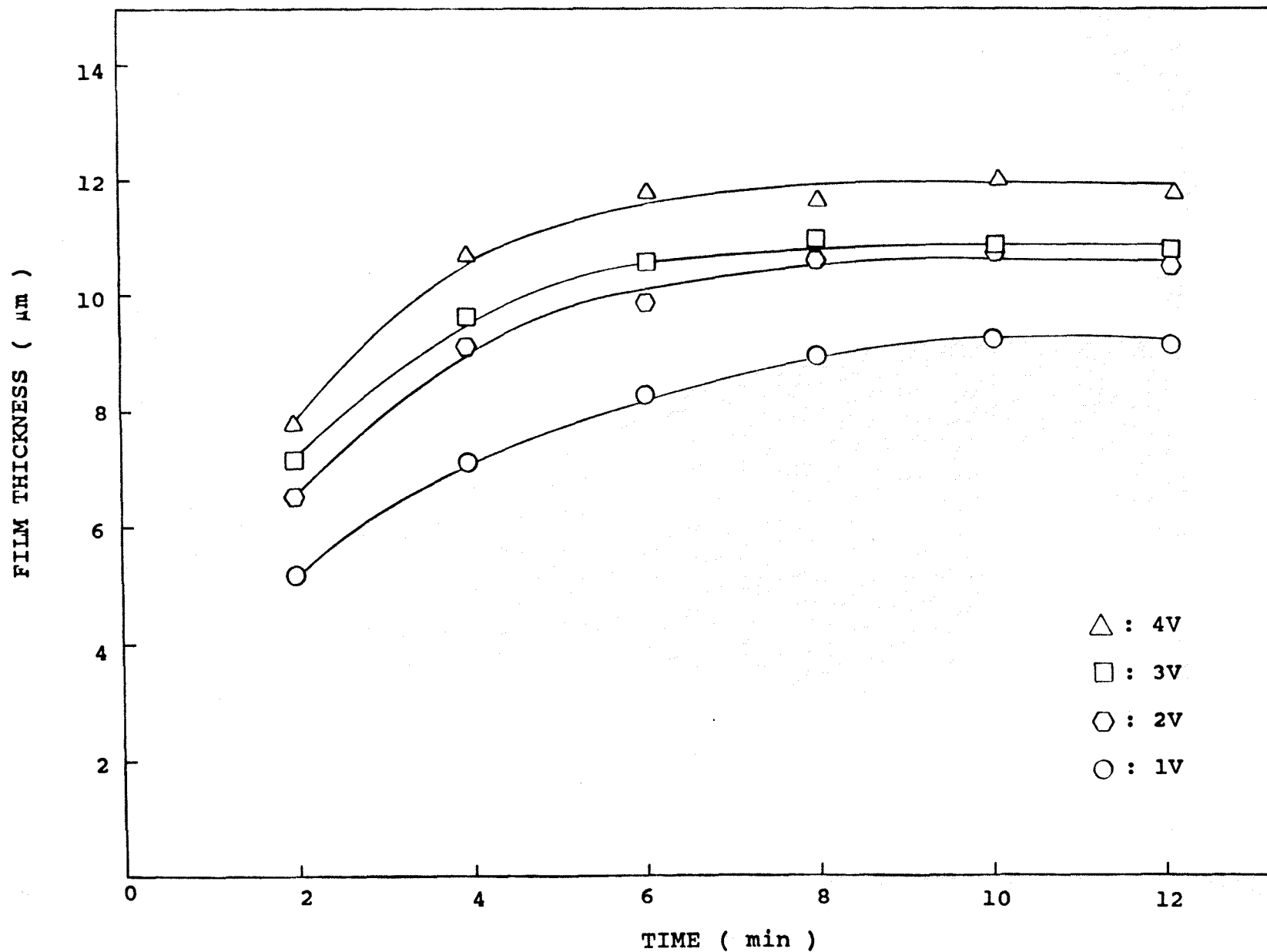


Figure 13: Variation of Film Thickness with Time for Constant Voltage Cathodic Electrodeposition using 25% PTFE Dispersion on Steel Surface

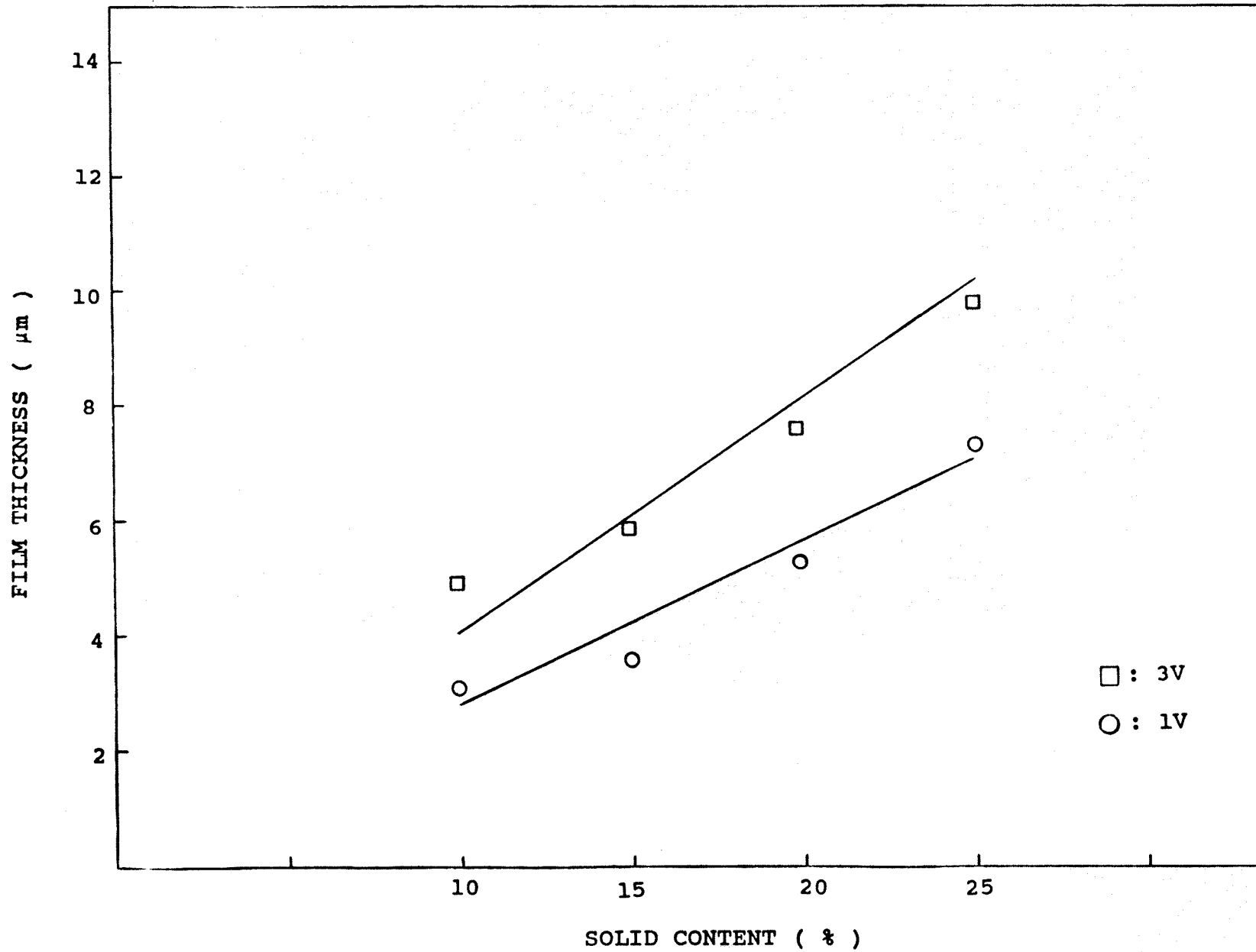


Figure 14: Variation of Film Thickness with Solid Content of PTFE Dispersion on Aluminum Surface at Time of 2 minutes

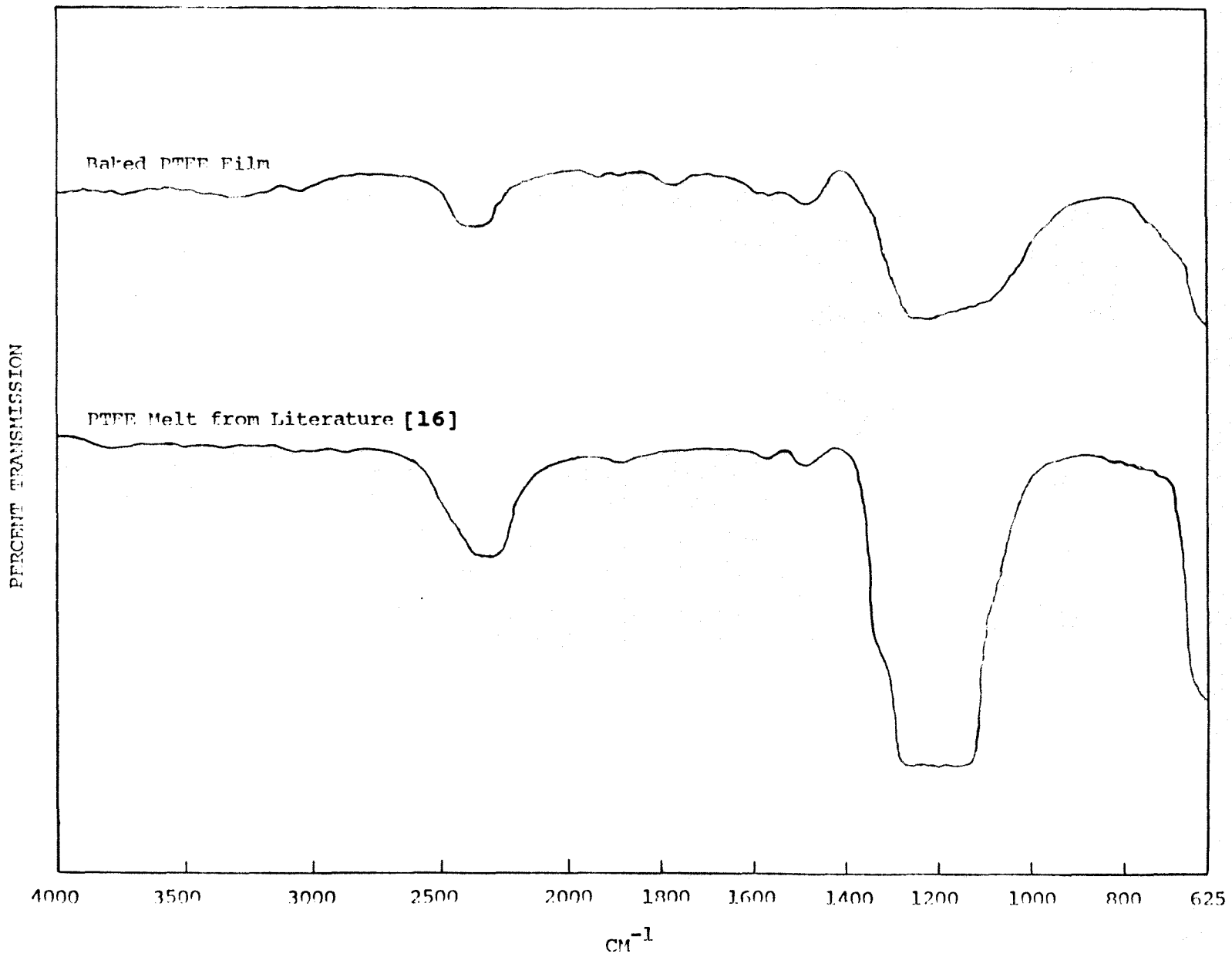


Figure 15: Infrared Spectra of Baked PTFE Film and PTFE Melt from Literature

Table 1: Composition of PTFE dispersions for cathodic electrodeposition powder coating process

Composition	Weight Percent (%)			
Polytetrafluoroethylene (PTFE)	8.00	12.50	17.00	21.50
Dodecylbenzyl dimethyl sulfonium chloride	0.65	0.85	1.05	1.25
Triton N-150	0.35	0.40	0.45	0.50
Acrysol GS	1.00	1.25	1.50	1.75
SGA 730	trace	trace	trace	trace
Ammonium hydroxide solution (6M)	trace	trace	trace	trace
Diisobutyl ketone	1.25	1.50	1.75	2.00
Water	88.75	83.50	78.25	73.00
Solid content (%)	10.00	15.00	20.00	25.00

Table 2: Weight and thickness of the deposited films from
10% and 15% PTFE dispersions on aluminum surface

Solid Content (%)	Deposition Time (min)	Applied Voltage							
		1 volt		2 volt		3 volt		4 volt	
		W ^a	δ ^b	W	δ	W	δ	W	δ
10%	2	18.7	3.04	27.1	4.40	27.7	4.49	36.5	5.92
	4	31.4	5.10	37.6	6.11	41.6	6.76	55.8	9.06
	6	42.4	6.89	43.2	7.02	50.6	8.21	61.3	9.95
	8	48.3	7.84	48.8	7.93	51.7	8.39	62.6	10.17
	10	46.8	7.59	49.6	8.05	50.8	8.25	61.5	9.98
	12	48.5	7.88	49.2	7.98	52.0	8.45	61.4	9.97
15%	2	21.8	3.54	24.8	4.02	35.8	5.81	42.2	6.85
	4	37.4	6.08	41.0	6.65	50.5	8.20	54.0	8.77
	6	47.4	7.69	50.4	8.18	55.9	9.07	63.1	10.25
	8	52.1	8.46	52.3	8.49	56.5	9.17	61.8	10.04
	10	56.1	9.11	54.0	8.77	56.7	9.21	62.3	10.12
	12	56.5	9.17	54.6	8.86	57.0	9.26	61.7	10.01

a: W is weight in mg.

b: δ is thickness in μm.

Table 3: Weight and thickness of the deposited films from
20% and 25% PTFE dispersions on aluminum surface

Solid Content (%)	Deposition Time (min)	Applied Voltage							
		1 volt		2 volt		3 volt		4 volt	
		W ^a	δ ^b	W	δ	W	δ	W	δ
20%	2	31.1	5.05	37.0	6.01	48.2	7.83	51.9	8.42
	4	49.2	7.98	47.4	7.69	62.6	10.17	67.9	11.02
	6	54.0	8.77	52.6	8.54	70.6	11.46	74.8	12.15
	8	61.7	10.02	60.7	9.85	69.3	11.25	75.5	12.26
	10	60.7	9.86	62.6	10.17	70.2	11.40	74.5	12.09
	12	61.3	9.95	62.8	10.20	69.8	11.34	75.0	12.18
25%	2	46.6	7.56	49.6	8.05	61.4	9.97	64.0	10.39
	4	56.0	9.10	60.8	9.87	74.5	12.10	83.2	13.50
	6	62.3	10.12	68.7	11.15	75.3	12.22	80.4	13.06
	8	67.1	10.89	73.7	11.97	75.1	12.19	84.9	13.78
	10	68.1	11.05	74.4	12.08	73.8	11.98	86.3	14.01
	12	67.8	11.00	74.2	12.05	74.1	12.03	86.0	13.96

a: W is weight in mg.

b: δ is thickness in μm .

Table 4: Weight and thickness of the deposited films from
10% and 15% PTFE dispersions on steel surface

Solid Content (%)	Deposition Time (min)	Applied Voltage							
		1 volt		2 volt		3 volt		4 volt	
		W ^a	ξ ^b	W	ξ	W	ξ	W	ξ
10%	2	13.4	2.17	20.9	3.39	25.9	4.20	30.3	4.92
	4	25.5	4.14	28.4	4.61	38.4	6.23	37.9	6.15
	6	31.2	5.07	35.4	5.74	44.2	7.17	47.3	7.68
	8	36.8	5.98	37.9	6.15	43.0	6.98	49.3	8.01
	10	37.7	6.12	40.1	6.49	44.3	7.19	49.0	7.96
	12	37.0	6.01	41.3	6.70	43.6	7.08	48.9	7.94
15%	2	17.9	2.91	24.8	4.04	30.2	4.90	33.2	5.39
	4	25.7	4.18	36.6	5.95	45.0	7.31	46.6	7.57
	6	35.8	5.81	39.8	6.47	43.7	7.12	50.1	8.12
	8	37.5	6.09	41.0	6.65	48.7	7.90	49.8	8.08
	10	36.8	5.98	41.8	6.79	48.0	7.79	50.1	8.14
	12	37.2	6.04	41.6	6.75	47.7	7.74	50.3	8.16

a: W is weight in mg.

b: ξ is thickness in μm .

Table 5: Weight and thickness of the deposited films from
20% and 25% PTFE dispersions on steel surface

Solid Content (%)	Deposition Time (min)	Applied Voltage							
		1 volt		2 volt		3 volt		4 volt	
		W ^a	δ ^b	W	δ	W	δ	W	δ
20%	2	24.8	4.02	27.8	4.51	37.4	6.07	41.2	6.69
	4	37.5	6.09	44.0	7.14	47.1	7.65	56.4	9.15
	6	46.4	7.53	49.5	8.03	56.1	9.11	62.6	10.17
	8	49.5	8.04	50.4	8.18	60.1	9.76	63.2	10.26
	10	50.5	8.20	50.3	8.16	59.8	9.70	62.5	10.14
	12	50.8	8.25	50.5	8.20	59.6	9.67	63.1	10.25
25%	2	30.6	4.97	38.3	6.22	43.5	7.06	48.8	7.93
	4	41.9	6.91	55.5	9.01	59.0	9.58	66.6	10.82
	6	49.8	8.09	60.9	9.89	65.7	10.66	72.9	11.83
	8	51.1	8.30	63.1	10.24	67.6	10.97	72.4	12.05
	10	51.8	8.41	63.4	10.30	66.4	10.78	74.2	12.05
	12	51.6	8.37	63.0	10.23	66.6	10.82	74.0	12.01

a: W is weight in mg.

b: δ is thickness in μm.