Investigation of thermal characteristic, surface morphology, mechanical properties and tribological properties of polyamide 11 powder coating

Poliamid 11 kaplamasının termal karakteristiği, yüzey morfolojisi, mekanik ve tribolojik özelliklerin incelenmesi

Yazar(lar) (Author(s)): Selim KEVRAN¹, Ömer Faruk MURATHAN²

ORCID¹: 0000-0001-9020-8523
ORCID²: 0000-0002-3636-2164

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Poliamid 11 Kaplamasının Termal Karakteristiği,
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Araştırma Makalesi / Research Article

Selim KEVRAN¹, Ömer Faruk MURATHAN²

¹Gazi Üniversitesi, Mühendislik Fakültesi, Kimya Mühendisliği Bölümü, Türkiye
²Gazi Üniversitesi, Teknoloji Fakültesi, Metalurji ve Malzeme Mühendisliği Bölümü, Türkiye

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ÖZ

Bu çalışmanın amacı, akışkan yatağa daldırma yöntemi ile uygulanan poliamid kaplamının mekanik ve kimyasal özelliklerinin incelenmesidir. Poliamid kaplama uygulaması için çelik test plakaları kullanmıştır. Spreyleme ile astar uygulanan test plakalarına, fırında ısıtma işlemi sonrası akışkan yataşta daldırma metoduyla poliamid kaplanmıştır. Kaplamanın mekanik dayanımının incelenmesi için aşınma dayanımı, çizilme testi, tuz testi ve sıcak yağa dayanım testleri yapılmıştır. Ayrıca kaplamanın termal karakteristiğini belirlemek amacı ile DSC, TGA analizleri yapılmıştır. Polimer kaplama ve astarın moleküler yapıları FTIR spektroskopisi ile analiz edilmiştir. Deneysel çalışma sonuçlarının gider kaplamada yaklaşık olarak %50 su bulunduğunu, kaplama yüzeyinin literatür araştırmalarına göre daha düzgün olduğu görülmüştür. Kaplamanın çizilme testi dayanımı 40N üzerindedir. Ağırlık testinde, 1,5kg yük kadar kaplama yüzeyinde herhangi bir çatlak görülmemiştir. Aşınma testinde 1kg ek yükleme kullanılmasına rağmen, 50.000 döngü sonrası poliamid kaplama iyi bir aşınma performansı sergilemiştir. Tuz testi ve sıcak yağ testleri sonrası kaplama yüzeyinde herhangi bir hata veya bozukluk gözlemlenmemiştir. Sonuç olarak akışkan yatağa daldırma metoduyla uygulanan poliamid toz kaplamalarını sert ve aşındırıcı çevrelerde ve mekanik dayanım gereklüğü mekanik parçalarda kullanılması uygundur.

Anahtar Kelimeler: Poliamid kaplama, termal karakteristik, yüzey morfolojisi, triboloji.

Investigation of Thermal Characteristic, Surface Morphology, Mechanical Properties and Tribological Properties of Polyamid 11 Powder Coating

ABSTRACT

The purpose of this study was to investigate the mechanical and chemical characteristics of polyamide coating by dipping in a fluidized bed system. For polyamide coating application steel test specimens were used. Test specimens, which were sprayed with primer, were cured in an oven and then coated by dipping in fluidized bed. To investigate the mechanical properties and abrasion resistance, scratch, salt spray and hot oil resistance tests were performed. Furthermore to investigate the thermal characteristics of coating DSC and TGA analyses were carried out. Molecular structure of polymer coating and primer were analysed by FTIR spectroscopy. Experimental results showed that primer consists nearly 50% percentage of water, coating surface was smoother than concerning investigations in literature. Scratch resistance of coating was above 40N. Up to 1.5kg load there was no crack on coating during falling weight test. Despite using an additional load of 1kg to abrasion wheels during 5000 cycles test polyamide coating showed fine abrasion performance. On the coating surface, there were no coating failure or deterioration failure was observed after salt spray and hot oil tests. In summary, polyamide powder coatings applied by dipping in a fluidized bed can be used in harsh corrosive environments and also this coating is suitable for mechanic parts where the mechanical strength is necessary.

Keywords: Polyamide coating, thermal characteristic, surface morphology, tribology.

1. INTRODUCTION

Powder coating products are environmentally friendly since volatile organic compounds (VOC) available in the liquid coatings; however they do not exist in the powder coatings. Powder coatings are based on polymeric resins. Thermoset and thermoplastic are main kinds of resins of powder coating. While epoxy, polyester and acrylic polymers are used as thermoset polymers; polyamide, polyethylene and polyvinyl chloride are mostly used ones for thermoplastic resin [1-3]. There are some advantages of polyamide powder coatings such as excellent impact and wear resistance, good corrosion resistance, hardness and low friction coefficient. Thanks to its coating characteristics, polyamide coating is used in different industries including automotive, textile, aerospace, mining.
Moreover, highly corrosive environment such as piping and oil industry, polyamide coating showed excellent corrosion resistance on steel parts [4-6].

There are two types of coating system mostly used in powder coating industry. They are fluidized bed system and electrostatic system [7-9]. However, some researchers investigated to use the fluidized bed and electrostatic power together. Barletta et al. investigated the influence of process parameters in an electrostatic fluidized bed coating system [10]. For fluidized bed system cleaning, curing temperature, dipping time, fluidizing and cooling condition has directly effect on coating physical and chemical properties. K.C. Leong et.al investigated the effect of dipping time, curing temperature on the coating thickness and roughness using polyamide and polyethylene powder coating [11].

Many studies have been carried with the aim to investigate the mechanical and chemical characteristic behaviors of polymeric coatings. Tian-xi Liu et.al searched the crystal transition of polyamide 11 nanocomposites by X-ray Diffraction [12]. Klm B.R. et. al and Giraldo L.F. et.al. used the micro-scratch test to estimate the scratch tensile strength of polymeric coatings [13,14]. Elsabee M.Z. et. al prepared several aromatic and aliphatic polyamide using an interfacial polymerization technique and then examined the crystallinity of these polyamides by X-ray diffraction, molecular structure by FTIR, thermal stability by Thermogravimetric analysis [15]. Martino, L.et. al prepared star shaped polyamide-11 samples by using aminoundecanoic acid from castor oil and analyzed the rheological and solid-state properties of these samples [16].

The objective of this work examines the mechanical and chemical characteristics of polyamide coated parts which were cured in an oven and then coated by dipping in the fluidized bed system. Although process seems simple, many parameters have an influence on final coating chemical and mechanical properties. The mechanical and chemical properties of polyamide-11 powder coating on steel test plates were investigated. For this purpose abrasion resistance test, scratch test, salt spray test and hot oil resistance test were performed. Moreover, to investigate the thermal characteristics of coating DSC and TGA analyses were carried out. Polymer coating and primer molecular structure was investigated by Fourier Transform Infrared Spectroscopy (FTIR-spectroscopy). Surface roughness and scratch strength of coating was estimated.

2. MATERIAL and METHOD

As a coating material, polyamide-11 powder was chosen. In order to increase the adhesion of polyamide powder on to metal surface, aqueous primer was used before application of polymer. St-37 steel was used as a test plate material in experiments. Various dimensions of test plates were decided according to test variables. Fluidized bed system in which coating process occurred was produced by using 3 mm stainless steel plate. As shown in Figure 1, fluidized bed was composed of 3 parts that are air intake zone, distributor and powder coating zone above nickel-chrome filter. During experiments, it was also monitored how fluidization occurred during powder coating of polyamide 11.

Coating steps are as follows; cleaning, drying, primer application, curing of primer and coating by dipping in fluidized bed system. The surface of the before coating directly affects the quality of coating, detailed cleaning procedures were applied to test panels. At first to remove soil, dust and oil test panels were dipped in alkaline solution for 15 minutes. Alkaline cleaner has 12 pH value and 70°C temperature. After for chemical etching, panels were dipped into 35% HCl solution for 1 minute. Before chemical etching, HCl acid inhibitor (Rodine57) was added to this acidic solution before dipping of test panel. Final treatment of cleaning procedure was cleaning panels with vapor degreasing solvent based on N-propyl bromide. Before each cleaning step, panels washed with de-ionized water. At the end of cleaning procedure panels were dried in oven at 70°C temperature for 1 hour.

Primer was applied by spraying to get a continuous and consistently film on each panel. Primer dry film thicknesses were measured between 8-15 µm. Curing conditions such as curing temperature and curing time were changed between 260°C and 310°C during 25 to 40 minutes to obtain different coating thickness. Test samples after curing process dipped in a fluidized bed between 3-5 seconds.

In order to determine the thermal characteristics of coating and primer TGA and DSC analyses were performed by TGA-Q500 model and DSC-Q2000 model instruments. For DSC analysis, aluminum pans
and for TGA analysis, platinum pans were used. DSC analysis powder coating material was performed by taking samples of powder which were nearly 11 mg.

Test was carried out by heating samples from -100 °C to 350 °C with heating rate of 10 °C/min. To get more accurate results, average values of analyses were taken. Also approximately 1.5 and 2 mg samples tested for TGA analysis, which were heated from 40 °C to 550 °C with heating rate of 20 °C/min.

DSC analyses of aqueous primer was performed by taking samples which were nearly 0.8 mg, 2.3 mg and 10.7 mg. Analyses were performed by heating samples from -100 °C to 350 °C with heating rate of 10 °C/min. Then approximately 3.5 mg and 1.7 mg samples tested with TGA, that the specimens were heated from 40 °C to 550 °C with heating rate of 20 °C/min.

In order to find out the microstructure of bonding interface between primer, coating and metal surface, SEM (QUANTA 400F) analysis were performed in Gazi University Photonics Research Center. St-37 steel plate was chosen as a test specimen. Test plate dimensions were 15x20 mm and thickness was 8 mm. The coating thicknesses of test plates were measured at three different points using a film thickness gauge (Dualscope MPOR - Fischer, Germany) and the average of this measurement was 150 μm. Cross section of the interface layers test specimens were scanned by SEM. SEM images were taken with different magnifications in order to get more accurate results.

In order to investigate the molecular structure of primer film and powder coating, Attenuated Total Reflection (ATR) analytical tool with the FTIR spectroscopy was used. ATR offers opportunity to examine various polymeric coating including very thin films without sample preparation [1]. Primer applied onto steel plate with spray gun and polymeric powder was analyzed with Bruker Vertex 80 FTIR spectrometer using ATR analytical tool. FTIR spectra were taken in the mid-infrared region between 4000-5500 cm⁻¹ wave number and 32 scans were averaged with the resolution of 4 cm⁻¹ at room temperature.

Coating surface roughness was measured by using Nanomagnetics-hp model Atomic Force Microscope (AFM). 10μm2 surface area was scanned at room temperature. For surface roughness test, 1.5x1.5 cm steel test plate was coated with polyamide-11 powder. To determine the scratch characteristic of coating, two scratch tests were applied by CMS model micro test scratcher with Rockwell 100 μm radius intender. First test was applied to 5 mm scratch length beginning 0.05 N to 5 N end loads by increasing load rate of 4.95 N/min. After first test we could not get any significant result so second test was applied to 6 mm scratch length beginning 0.05 N to 25 N end loads by increasing load rate of 24.95 N/min. Since maximum load capacity of micro test scratcher was 25 N, another test scratcher with maximum load capacity of 40 N (Sheen Instrument) was used.

The hardness of coated test panels were measured according to ISO 868 standard and shore hardness test was applied with using a durometer (LD0550 Model -TQC Netherlands). Moreover, to determine the hardness of coating, pendulum hardness test applied to test specimen according to EN ISO 1522 standard with BYK model pendulum hardness tester. 1mm thick St-37 steel plate coated by polyamide-11 (having 130μm thickness) was used for pendulum hardness test.

The impact resistance of coating was investigated using Falling Weight Impact Test according to ISO 6271-1 standard. Tests were performed with a tubular impact tester (SH807 Model, Sheep, UK) dropping ball with variable mass up to 2 kg from 1 m height. The test results were scrutinized with a 5X magnifying glass. The critical load and height was found when coating failure was seen. For these parameters impact force and kinetic energy of ball just before the impact was calculated. Net potential energy of mass equal to kinetic energy, and this energy is equal to work done after impact by mass. Penetration depth of mass after impact was measured as 3 mm when there is no coating crack on test panel.

Abrasion resistance of coating was investigated by using a taber abraser (Qualitext, USA). Tests were conducted by using a sand wheel with 1000 g load and test period was 5000 cycle of wheel with velocity of 72 r/min. For abrasion resistance, spherical steel plate with a diameter of 10 cm was used. The coating thickness of test plate was measured at the beginning and at the end of the tests in order to evaluate abrasion resistance.

The corrosion resistance of coating was evaluated with a salt spray test chamber (SPP Model, QLAB, USA) according to ISO 9227 standard. Test panels placed on an inclined surface of an angle 20 degree with vertical and 5% NaCl solution was added to chamber. Chamber inside temperature was adjusted to 35°C and atomized solution was sprayed to cabinet continuously during the test.

For determination of resistance to hot oil tests were carried out according to ISO 2812 standard. At the end of salt spray chamber test, coated plates were dipped in 10w40 engine oil at 60°C temperature for 168 hours in order to find out both the corrosion and thermal resistance of coating.
After cleaning steps, test specimen was examined by visible inspection for coating failure. Experimental test parameters are given in Table 1.

| Test Specimen Material | St-37 | St-37 | St-37 |
|------------------------|-------|-------|-------|
| Dimensions of Test Specimen (cm) | 15x7x0.1 | 15x7x0.1 | 15x7x0.1 |
| Curing Temperature (°C) | 280 | 290 | 310 |
| Curing Time (min.) | 25 | 30 | 40 |
| Dipping Time (sec.) | 3 | 3 | 3 |
| Primer Thickness (mm) | 13 | 15 | 10 |
| Polyamide-11 Coating Thickness (mm) | 150 | 180 | 230 |
| Salt Spray Test Period (h) | 1000 | 1000 | 1000 |

3. RESULTS AND DISCUSSION

3.1. Thermal Characteristics

In the DSC spectrum of the primer peaks were observed at about 0°C and 100°C (Fig.2). These temperatures correspond with the phase change temperatures of water so DSC spectrum verified the presence of water in primer. The calculated enthalpy values from the graph are 166 J/g and 1250 J/g respectively. These values are nearly half of the fusion and vaporization enthalpy of water as expected because the weight ratio of water per gram of primer is nearly 50% from the MSDS of primer.

In Figure 4, there are two curves on the graph at 100°C and 395°C. The curve at 100°C is observed because of the vaporization of water in the primer and 50% weight loss verified the DSC result of primer.

Thermal decomposition of primer started at 380°C and ended at around 430°C (Fig.5). Weight loss is nearly 40% and remaining 10% is residual ash content of primer.
Thermal decomposition of polymer started at 390°C and ended at around 440°C. Beyler et al., [19] also found that gelation and discoloration of polyamides start at about 400°C.

3.2. Surface morphology
To analyze the bonding interface between primer, coating and substrate; coated samples were scanned by SEM at different viewing scales. SEM images showed substantial bonding between metal and primer surface. As shown in Fig. 6 it is obviously seen how primer adjust metal surface for further bonding to coating due to the smooth surface.

Figure 6. SEM images of the primer/substrate interface with different magnifications a) X750 and b) X100

Between acid etched metal surface and primer surface there was no bonding failure or separation. Moreover, from the cross sectional SEM images of metal and coating surface in the Fig.7, it is clear that polyamide coating adhered to the metal-primer surface excellently. The coating thickness of test samples which were measured by thickness gauge, nearly 150 µm and this value was also verified by SEM images.

Figure 7. SEM images of the coating/substrate interface with different magnifications a) X50, b) X100 c) X250 and d) X400

3.3. Chemical composition and structure analysis
FTIR spectra of polymer show a peak at around 3298 cm\(^{-1}\) and a weak peak also is observed at 3080 cm\(^{-1}\) due to the N-H stretching (Fig.8). The –CH and –CH\(_2\) stretching vibrations show two sharp peaks at around 2919 cm\(^{-1}\) and 2850 cm\(^{-1}\). On the FTIR spectra the sharpest peak appear at 1637 cm\(^{-1}\) due to the C=O stretching which is a characteristic peak of amide group. Strong peak seen on the spectrum at 1535 cm\(^{-1}\) is due to N–H bending and C–N stretching because these stretchings usually give peak at around 1540 cm\(^{-1}\). Two peaks appear at 1466 cm\(^{-1}\) and 1352 cm\(^{-1}\) are due to CH\(_2\) scissoring and CH bending respectively. The FTIR spectra of polyamide-11 (Fig.8) almost exactly coherent with the spectrum of polyamide-6 given in literature [20-22].

Figure 8. FTIR spectra of polymer powder

FTIR spectra of primer shows a broad weak peak at 3271 cm\(^{-1}\) due to the N-H stretching and CH\(_2\) stretching show peak at 2926 cm\(^{-1}\) (Fig.9). Carbonyl stretching (C=O) peak appear at 1726 cm\(^{-1}\) and C=C bond show peaks at 1605 cm\(^{-1}\) and at around 1500 cm\(^{-1}\). C–O bond gives peak between 1000 cm\(^{-1}\) and 1300 cm\(^{-1}\). On spectrum of primer three peaks at 1221.1178 and 1009 cm\(^{-1}\) are due to the C–O bond. The =C–H out of plane bending band is observed at 824 cm\(^{-1}\).

Figure 9. FTIR spectra of primer

3.4. Surface Morphology
10x10µm\(^2\) area of polymer coating was scanned with AFM (Fig. 10). Analyzed test specimen’s coating thickness is measured as 140 micrometer. Maximum height (height between the lowest recesses and the highest projection) on scanned area is 0.98 µm.
The coating roughness parameters are given in Table 2. Farshad et al. [23] measured the average roughness value of Nylon coating as 0.04 µm with AFM and 0.45 µm with a profilometer and they also measured the distance between peak and valley of nylon coating as 0.32 µm with AFM and 1.07 µm with a profilometer. Their roughness values are almost same with our measured roughness values. Al-Hadharami et al. were also measured the average roughness value of their phenolic epoxy based polymeric coating as 0.43 µm [24]. From the results of another study, the average roughness value of polyolefin based thermoplastic coating was measured as 1.2 µm [25]. According to the literature the surface of our coating was at least as smooth as others.

### 3.5. Mechanical Properties

Table 3 shows the hardness values of the final coating on St-37 test specimen. 30 µm increased coating thickness has no effect on the shore hardness value. According to polymeric materials the coating hardness can be acceptable.

| Coefficient | Description     | Value | Unit |
|-------------|-----------------|-------|------|
| sa          | Average         | 0.148 | µm   |
| sq          | Root Mean Square| 0.177 | µm   |
| ssk         | Surface Skewness| 0.09  |      |
| sku         | Surface Kurtosis | 2.37  |      |

To find out the scratch strength of the coating, micro scratch test technique and mechanical scratch tests were applied. While performing micro scratch test, constant increasing rate of load was applied to coated test panels. With progressive 25 N load, any critical load could not be detected.

As seen in Figure 11 and Figure 12, maximum frictional force and maximum penetration depth are 10 N and 130 µm respectively. Frictional force and penetration depth increased almost linearly with applied load. It can be seen from the curves that polymers showed less fluctuation and smooth changes during test because of its visco-elastic behavior just as investigations related with this issue in literature [26-27].
Coated panels were scratched with Sheen model tester and three different load applied on test panels (Fig.13). For all loads it could not be seen the bare metal beneath the coating and coating so scratch resistance of polyamide 11 coating was determined above the 40N.

120μm thickness of polyamide coated test panels were analyzed for the impact resistance of coating by falling weight impact test. First, 1kg weight was dropped from 1m height on to the test panel and there was no crack on coating. Then 1.2 kg weight was dropped and it was not seen any crack on coating. After 1.5 and 2 kg weights dropped there were coating cracks on test panels (Fig.14). Penetration depth was measured as 3mm and so impact force and kinetic energy before impact were calculated as 3920 N and 11.76 J respectively.

![Figure 14. Falling weight impact test results of 120 μm polyamide coated test panels with various loads](image)

Evaluation of abrasion test can be done by many ways such as; weight index, weight loss and wear cycles per mil. After the test we chose wear cycles per mil to evaluate the wear resistance of coating (Fig.15).

![Figure 15. Abrasion test of polyamide coated test specimen after 5000 cycle under 1 kg load](image)

In order to find out the corrosion resistance of polyamide coating under harsh conditions, salt spray and hot oil tests were performed respectively to the same test plates. Figure 16 and Figure 17 shows the corrosion performance of polyamide coating under salt spray and hot oil tests and it can be seen that there is no deformation or corruption on the coating surface. Also, the resistance of coating to salt spray and hot oil test was good for all different thickness level of coatings. Any coating defect and failure was not observed on test specimen after physical examination with magnifying glass.

4. CONCLUSION

In the present study, polyamide-11 powder coating and aqueous primer were applied on the metal test specimens by using fluidized bed reactor. Related to coating process, the increase of curing time and curing temperature increased the thickness of coating. Microstructure, mechanical properties, surface morphology and tribological properties of coating were investigated. The glass transition and melting temperature of polyamide coating were determined as 46 °C and 186 °C respectively by DSC analysis. DSC analysis of primer also showed that primer contain water nearly 50 % weight for percentage. Thermal decomposition temperatures of primer and polyamide
coating were estimated at 395 °C and 421 °C by TGA analysis. The average surface morphology value measured by AFM confirmed that coating surface is at least as smooth as the concerning investigations in literature. It was found that scratch resistance of coating is above 40 N. Up to 1.5 kg load there was no crack on coating during falling weight test. Despite using an additional load of 1 kg to abrasion wheels during 5000 cycles of test polyamide coating showed fine abrasion performance. At the end of salt spray and hot oil tests any coating failure and deterioration were not observed on the coating surface. In summary, polyamide powder coating that was applied by fluidized bed reactor can be used in harsh corrosive environments. In addition, it is suitable for mechanic parts when considered its mechanical strength.

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