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THE EFFECTS OF UV EXPOSURE ON THE STRUCTURAL AND MECHANICAL PROPERTIES OF BNNSs THIN FILMS

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Abstract
Boron nitride nanosheets (BNNSs) are good candidate for UV protection. They have ability to absorb the harmful UV part of sunlight and prevent the coated substrate against the environmental degradations. There are several studies focused on boron nitride coatings on copper, steel or etc. for UV protection. However, studies on boron nitride coating on the glass surface are scarce and the behavior of nanostructured thin BNNSs films and long-term effects of UV-radiation exposure have not been studied yet. In this study, we report the effect of UV radiation on the structural, physical and mechanical properties of BNNSs thin films. Boron nitride nanosheets were obtained using isopropyl alcohol (IPA) as solvent with liquid exfoliation method. BNNSs thin films were produced using different immersion numbers at a dipping rate of 3 mm/s (5,10, 20, 30, 50). Obtained thin films were evaluated for the UV protection ability after 15 and 25 hours as exposure time. Scanning electron microscopy (SEM), UV-visible spectroscopy, was carried out to study the structure and optical properties of the BNNSs films as a function of the UV-irradiation time and immersion number. Mechanical properties of thin films were obtained using 3-point bending test. Atomic Force Microscopy (AFM) was conducted to determine the thickness of nanosheets. It was observed that the thickness of nanosheets is generally changed between 1-4 nm. As the number of immersions increased, the transmittance of the glass surfaces decreased.

Keywords: Two-dimensional nanomaterials, BNNSs, transparent BNNSs thin film, UV protective materials, dip-coating method.

1. Introduction
BNNSs, referred to as "white graphenes," are composed of several layers of hexagonal BN planes and have unique properties such as a wide energy band gap, electrical isolation, ultraviolet (UV) photoluminescence, high thermal conductivity, and stability. BNNSs are (002) mono/several layer hexagonal BN (h-BN) and are structural analogues of graphenes. h-BN is a widely used compound with equal amounts of boron and nitrogen atoms, in skin care products, insulators, solid lubricants, lead pencils and so on. BN crystal cages are similar to graphene, but with a stronger B-N bond, which gives them superior properties in mechanical and thermal applications. On the other hand, since B has a significant charge transfer to N atoms, ionic sp^2 hybridized B-N bonds have exhibit considerably different properties optically and electronically. BNNSs are valuable insulators for dielectric applications, for example they can be used as a dielectric barrier layer and a material with deep ultraviolet light [1]. Synthesis of BNNS is one of the less well-researched topics for both scientific research and industrial applications, and studies have opened the way for large-scale synthesis of high-quality BNNS. The main methods of obtaining boron nitride nanosheets are liquid exfoliations, chemical vapor deposition, the opening of boron nitride nanotubes, and solid-state reactions. Recently, different methods have been used to obtain BNNSs. Liu et al. used boron nitride nanosheets in hexagonal structure as a high-performance flame-retardant coating. In their exfoliating process, 4 g of bulk BN powder was dispersed in a Sodium dodecylbenzenesulfonate (SDBS)/water solution. Here, the SDBS concentration was fixed at 1 mg/mL. The BN/SDBS/water distribution was sonicated for 4 hours with 3 seconds open and 3 seconds closed. Suspension is kept in the ice bath to suppress the generated heat. After 4 hours of sonication, the suspension was kept free overnight and over 80% of the supernatant was collected. It was then centrifuged at 1500 rpm for 90 minutes. After this process, 60% of the supernatant was collected at the top of the suspension and the remaining residue was discarded. As a result, Liu et al. have proved the use of exfoliated h-BN nanosheets as fire resistant coatings. After the fire was exposed, the surface of the wood material remained undisturbed [2]. Another study on boron nitride nanosheets was conducted by Marsh et al. The suspension of hexagonal boron nitride was prepared using an auxiliary solution. This system combines the common organic solvents with water to form a mixture that melts and suspends h-BN in a more efficient manner in the separate components. Solvents were chosen based on molecular weights (M.W.), boiling point, ease of use and safety considerations. Based on these criteria, methanol (MeOH), ethanol (EtOH), 1-propanol (1-prop), 2-propanol (IPA), acetone and tert-butanol (tBA) were chosen. BNNS was prepared by ultrasonication method. Bulk h-BN powder was added to a co-solvent mixture at a loading of 2 mg/mL. The suspension was then sonicated in a bath sonicator for 3 hours, rotating the sample bottles every 30 minutes to ensure the most homogenous mixing possible. The samples were then centrifuged at 3200 rpm for 20 minutes, and the residue on the suspension was collected for analysis. They stated that a 60% w/w TBA mixture in water is most effective to form a stable BNNS suspension. TEM images confirm the exfoliation of the layers [3]. Yurdakul et al. investigated the production of BNNSs

by microfluidization method. A high pressure microfluidizer fluid processor was used for the first time to exfoliate few layers two dimensional BNNSs using micro-sized h-BN as a precursor. A mixture of N,N-dimethylformamide and chloroform was used as solvent. High pressure microfiltration was performed with a commercially available microfluidizer (M-110P, Microfludics Corp.) to produce chemically exfoliated BNNSs. It was observed that BNNSs had micrometer dimensions in the plane, and it was seen that the nanometer dimensions along the thickness of the layers were dependent on the number of layers stacked together. It was calculated that the thickness of 2D BNNSs was between 8 and 12 nm using EELS analysis. This value suggests that the BNNs are composed of about 20 to 30 monatomic h-BN layers [4].

Recently, the extermination of the ozone layer in the earth's atmosphere has caused to an increased risk to environment, especially to plant, human and animal life. Since ultraviolet (UV) radiation triggers the formation of free radicals, the long-term exposure can lead to damage for human skin, food products, materials etc. So, several products must be protected to long periods under UV radiation [5]. UV absorbers have been used to reduce these harmful effects and to ensure that the properties of the materials are adequately protected. To provide protection against UV radiation, one of the UV-absorbing molecules is the ability to reduce the combined energy of the radiation to a thermal energy that provides less energy through a photophysical process [6]. Inorganic materials such as metal oxide semiconductors can effectively absorb UV radiation and show good heat resistance properties. There are several inorganic compounds which have UV protective properties such as TiO_2, CeO_2, ZnO [5]. Some of these compounds cause coloring, thus limiting their use. BNNSs are an alternative powerful UV preservative to these inorganic compounds. There are many studies in the literature about UV protective coating on glass substrate. Tran et.al. investigated the functionalized hexagonal boron nitride nanocoatings for protection of transparent plastics. In order to increase hardness and water resistance and decrease the UV degradation level of coatings and transparent plastics h-BNs were modified with [3-(2-Aminoethylamino) propyl] trimethoxysilane and uniformly dispersed into the polyurethane coatings adding several amounts, such as 0.1, 0.2, 0.4, and 0.8wt%. The coated samples were analyzed by Fourier Transform Infrared Spectroscopy (FTIR), UV-Vis Spectroscopy, Scanning Electron Microscope (SEM), Water Contact Angle, and Differential Scanning Calorimeter (DSC). The test results indicated the nanocoatings with functionalized h-BN showed superior physical and chemical behaviors against the UV and other physical degradations on the substrates. Researchers believe that this nanocoatings will also increase the corrosion resistance of other metallic surfaces [7]. Gao et al. have studied high-yield synthesis of boron nitride nanosheets with strong ultraviolet cathodoluminescence emission. Bulk quantities of hexagonal boron nitride (h-BN) nanosheets have been synthesized via a simple template and catalyst-free chemical vapor deposition process at 1100-1300 °C. IR spectrum and electron energy loss spectra show the typical nature of sp^2-hybridization for the BNNSs. Under the same conditions, the initial oxidation temperature for carbon nanotubes is 400°C, while for BN nanosheets there is an initial oxidation temperature of 850°C. This demonstrates that h-BN nanosheets, which exhibit strong ultraviolet laser behavior, emit strong and narrow cathodoluminescence emissions in the ultraviolet range. Due to the insensitivity of this luminescent response, the BN nanosheets are highly ideal for the laser processing of optical devices in the UV regime [8]. When literature researches are examined, it has been seen that UV protective coating for glass surfaces has not been studied before. Researches carried out to date have generally been related to UV absorber materials incorporated into the glass structure. Thanks to this work, it is aimed to protect the light and heat sensitive products in the glass packaging with the coating to be applied on the glass in different usage areas. For this reason, the glass surface coated with BNNSs at different immersion times was exposed to UV light for various periods of time, and the protection of the glass surface was determined by various physical and mechanical tests.

2. Materials and Method

2.1. Materials

Boron nitride as nanosheet starting material (2.27 g/cm^3,>98% purity, micron size, Bortek Boron Technologies and Mechatronics Ind. Co.) and isopropyl alcohol (J.T. Baker) as solvent were used to prepare immersion solution in the experiments. Polyvinyl butyral (PVB, ABCR in 0.1% by weight isopropyl alcohol solution) was used to increase the adhesion of the boron nitride to the glass surface and to facilitate its adhesion. Ethanol (99% pure, Sigma Aldrich) and distilled water was used to clean the surface and increase wettability of glass. Borosilicate microscope slides (25.4x76.2 mm in size and 1-1.2 mm in thickness, ISOLAB) were used as the surface to be coated.

2.2. Synthesis of BNNSs

A schematic diagram of the synthesis of the boron nitride nanosheet is shown in Figure 1.

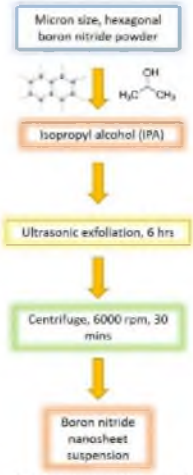

Figure 1. Preparation of BNNSs

Isopropyl alcohol was used as solvent in this method. 0.2 g of bulk boron nitride is dispersed in 100 mL of solvent (2 mg/mL). The resulting mixture was sonicated for a total of 6 hours at 45 minutes intervals in an ultrasonic bath (ISOLAB) at a power of 180 W and centrifuged (Centurion, C2 Series) for 30 minutes at 6000 rpm to complete the formation of the boron nitride nanosheets. The supernatant was taken to a new bottle. This suspension was used as a dipping suspension to form a thin film of boron nitride nanosheet.

2.3. Formation of BNNSs Thin Films

The glass surfaces to be coated in thin film formation are washed first with detergent water. Glass surfaces were immersed in distilled water and then washed in ultrasonic bath for 15 minutes with ethanol to avoid any impurities and contamination. The cleaned glass surfaces were left to dry in the environment. Completely dry and clean surfaces were first dipped into the PVB solution 3 times at 3 mm/s using a dip coating machine (ETOKS Electronics and Software Inc.) to increase the adhesion between the material to be coated and the glass surface. Subsequently, at the same rate, the prepared BNNSs solution was submerged in different dipping numbers (5,10, 20, 30, 50). The glass surfaces were heated to 400°C for 2 hrs in the ash furnace (Protherm) so that the PVB is partly removed from the glass surface after the immersion process and the boron nitride remains as a single layer on the glass surface. Table 1 gives the experimental working conditions for the formation of boron nitride nanosheet thin films.

Table 1. Experimental operating conditions for the formation of thin films of boron nitride nanosheet

Sample Code	PVB Immersion Number	BNNSs Immersion Number	Speed of dipping and withdrawal (mm/s)	Heat Treatment Temperature (°C)	Heat Treatment Time (Hours)
53	3	5	3	400	2
103	3	10	3	400	2
203	3	20	3	400	2
303	3	30	3	400	2
503	3	50	3	400	2

2.4. UV Exposure of BNNSs Thin Film

The samples were irradiated with a UV-A 340 lamp for 15 and 25 hours. The Atlas UV Test machine was used to provide UV exposure depending on time. The measured light power reaching the sample was 1.45 W/m^2 in the UV-A range, mainly responsible for the degradation of the coated and uncoated sample. UV exposure test conditions were given in Table 2.

Table 2. UV exposure conditions of BNNSs thin films

Sample Code	UV Exposure Time (Hours)
53-15	15
103-15	15
203-15	15
303-15	15
503-15	15
53-25	25
103-25	25
203-25	25
303-25	25
503-25	25

2.5. Characterization

Morphological image of boron nitride nanosheet thin films was investigated by SEM (Zeiss Supra 40VP, Germany) analysis at different magnifications. Film transmittance before and after UV exposure were determined using UV visible region spectroscopy (Agilent Technologies, Cary 60 Uv-Vis). Thickness of boron nitride nanosheets was obtained with Atomic Force Microscopy (Parksistem). The mechanical properties of the glass surfaces as a result of the coating and UV exposure were determined using a 3-point bending tester (Shimadzu AG-IC Test Machine) at a loading speed of 2 mm/min.

3. Results and Discussion

3.1. AFM Images of BNNSs

The thickness of the boron nitride nanosheets and surface images were determined using Atomic Force Microscopy. The images of the nanosheets were taken from the portion of the silicon wafer surface obtained by immersing the wafer surface in the immersion suspension once. Figure 2 shows the thickness of nanosheets from different regions on the surface.

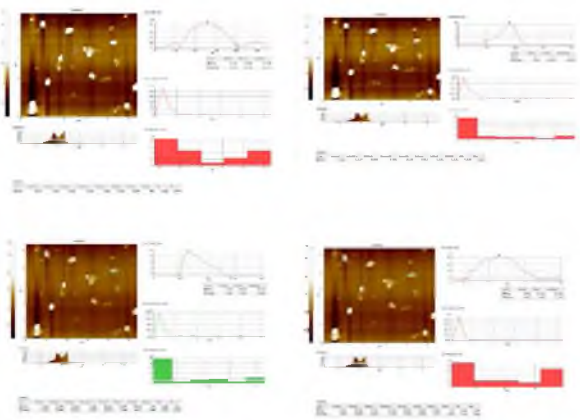

Figure 2. Thickness measurement of boron nitride nanosheets

Figure 2 shows the thickness measurement of nanosheets from different regions. The thickness of nanosheets varies from 1-4 nm. When looking at the two histograms in the upper section it can be indicated that the thickness of the nanosheets varies from 1-4 nm in general. Light-colored nanosheets represent thinner layers, while darker colored parts denote thicker layers. In the last two images, measurements were taken from darker colored nanosheets. The thickness of the nanosheets in this section ranges from 4-10 nm. Because of the observation of thick nanosheets, a single layer coating on the silicon wafer results in an agglomeration overlying nanosheet. A thickness of 2 nm represents about 5 layers [9]. For this reason, it can be said that the nanosheets consist of several layers if the first two images are taken as basis. Studies conducted in the literature show that the thickness of boron nitride nanosheets generally varies between 2-5 nm [10-12]. The lateral dimension of nanosheets varies between 200-400 nm.

3.2. SEM Images and EDX Analysis of BNNSs Thin Films

Figure 3 contains SEM images before and after UV degradation.

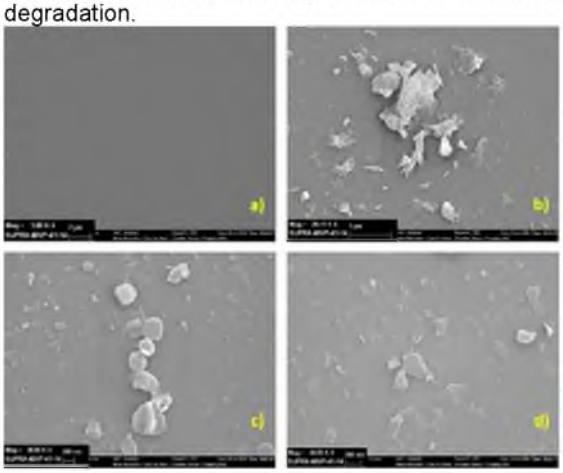

Figure 3. SEM images of thin films a) pure glass b)503, c) 503-15, d)503-25

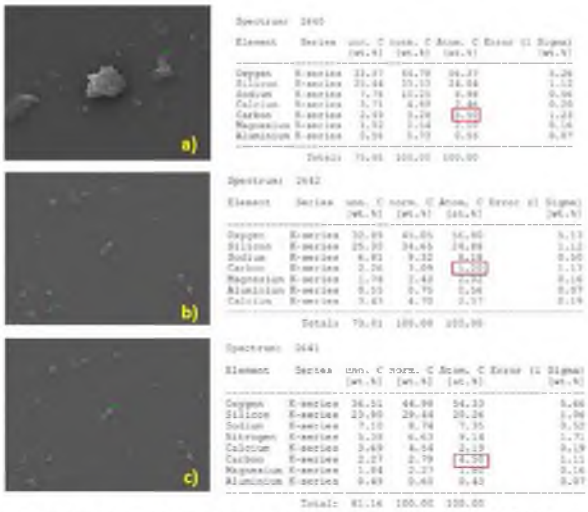

Figure 4. EDX analysis of thin films a)503, b) 503-15, c)503-25

When the images were examined, the structure of the boron nitride nanosheets interacted with PVB in the coatings prior to the UV decomposition (Figure 3-b). 15 and 25-hour UV degradation caused the PVB to be removed partly from the structure, leaving the structure of the boron nitride nanosheets intact. This situation is also parallel to EDX results (Figure 4). The carbon content decreased from 5.50% to 5.20% after 15 hours of UV degradation and to 4.50% after 25 hours of UV degradation.

3.3. Optical Transmittance Results of Thin Films

Transmittance analyzes of thin film coatings were carried out using a UV-Visible Spectrophotometer at a wavelength range of 100-800 cm^{-1}. Figure 5 contains the results of the transmittance of thin films at different immersion numbers, as well as the transmittance of the PVB coated glass surface and the uncoated glass surface prior to UV degradation.

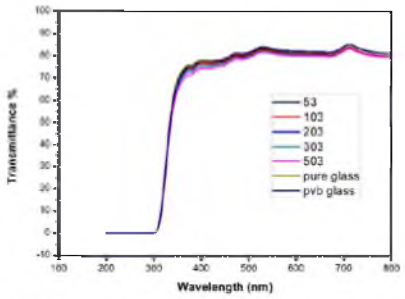

Figure 5. Optical transmittance results of thin films obtained at different immersion numbers before UV exposure

When the transmittance results are examined, it is possible to say clearly that the transmittance decreases as the number of immersion increases. As the number of immersion increases, a darker film is obtained, so the transmittance decreases. In the study conducted by Pereira da Silva et al., zinc oxide: aluminum thin films were coated on glass surface in different thicknesses and observed that

optical transparency in the visible region decreased considerably as the coating thickness increased. This reduction in optical transmission in the infrared region was associated with an increase in light reflection due to the increased load-carrier density [13]. When Figure 5 is examined, it is observed that the permeability in the same wavelength (400 nm) with respect to the uncoated glass is reduced by about 12.5% by plating 50 times. 20 and 30 times, similar permeability results were obtained in the coating, the permeability decreased by 10% compared to the uncoated glass. Coatings made by dipping 5 and 10 times were also similar in permeability and their permeability was reduced by about 6.25% compared to the uncoated surface. Figure 6 shows the optical transmittance results of thin films after UV exposure at 15 and 25 hours.

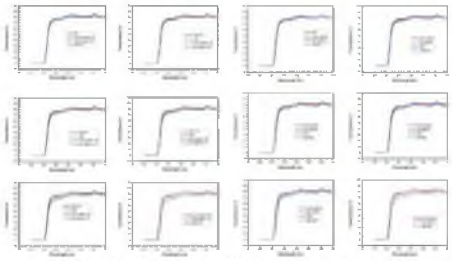

Figure 6. Optical transmittance results of thin films obtained at different immersion numbers after 15 and 25 hrs UV exposure

Each graph shows the optical transmittance results after 15 and 25 hours UV decay, which occurs at different dipping numbers. When the graphs were examined, it was observed that the transmittance decreased after UV degradation in all plated glass samples. The most noticeable decline was seen after plated by dipping 50 times. This reduction can be attributed to the protective property of the boron nitride nanosheets in the UV-A region.

3.4. Mechanical Properties of BNNSs Coated Glass

The 3-point bending test was performed to determine the change in the mechanical properties of the coated glass relative to the uncoated glass and to observe the effects of UV degradation on the glass bending strength and elastic modulus. The 3-point bending test was performed using a 40 mm inter-jaw distance condition at a loading rate of 2 mm/min. Figure 7 shows the flexural strength and flexural modulus of BNNSs coated glass before and after UV degradation for 15 hours. The bending strength of the uncoated glass and the glass obtained only by PVB coating is similar and is about 116 N / mm^2. Though there was no significant change in the bending strength of the thin-film glass coated 5 times, it was observed that the bending strength of the glass decreased when the number of immersions was increased. In particular, the bending strength of the glass obtained by immersion for 10 times was about 105 N/mm^2, which decreased by 25.71% for 20 times as a result of immersion. The most significant decline was observed in this range. The bending strengths of the glasses obtained by dipping 30 and 50 times did not change much compared to dipping 20 times. Dipping 5 times did not bring about a change in the bending strength of the glass. Different models have been developed to describe increased strength in thinner films. The Nix model describes the dislocation effects resulting from channeling along the thickness. In response to the applied stress, the pulling segment of the strip moves forward to reduce the tensile energy, while the accumulated dropout segment becomes longer, resulting in more energy expenditure. This leads to a threshold stress for the dislocation movement and consequently the driving force causes the tension to move with the opposite of the film thickness to reduce this accumulated release segment. When the particle size is similar to the thickness, the particle size decreases with decreasing film thickness. This dislocation, which is also due to displacement due to accumulation in the grain boundary, is strengthened by Hall-Petch effect. This shows that the strength of the film increases with decreasing film thickness [14].

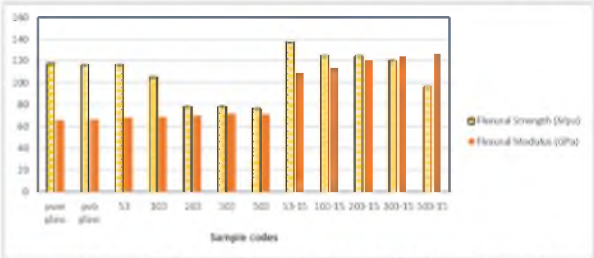

Figure 7. Mechanical properties of BNNSs coated glass before and after 15 hrs UV exposure

When the results were examined after 15 hours of UV degradation, the bending strength of the glass showed an increase with respect to the previous state without degradation, and accordingly, the decrease in the elastic modulus of the glasses was observed by increasing the number of immersions. The bending strength of the glass coated with 5 times of dip showed an increase of 6.5% while 10, 20 and 30 times immersion, showing a 15% increase after 15 hours of UV degradation. After 25 hours of UV degradation, the bending strength of the 5 and 10 times dipped glass showed a decline compared to the previous state without degradation (Figure 8). In the case of coatings made more than 10 times, almost no change was observed compared to the situation before the UV decay.

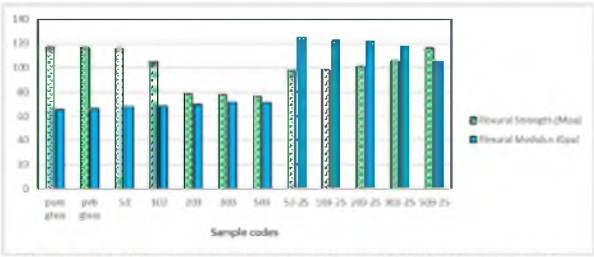

Figure 8. Mechanical properties of BNNSs coated glass before and after 25 hrs UV exposure

4. Conclusions

- The thickness of the nanosheets ranges from 4-10 nm. Because of the observation

of thick nanosheets, a single layer coating on the silicon wafer results in an agglomeration overlying nanosheet.

- The structure of the BNNSs interacted with PVB in the coatings prior to the UV decomposition. 15 and 25-hour UV degradation caused the some of PVB to be removed from the structure.
- Optical transmittance decreased as the number of immersions increased. The most noticeable decline was seen after plated by dipping 50 times. This reduction can be attributed to the protective property of the boron nitride nanosheets in the UV-A region.
- After 15 hours of UV degradation, the bending strength of the glass demonstrated an increase with respect to the previous state without degradation, and accordingly, the decrease in the elastic modulus occurred by increasing the number of immersions. After 25 hours of UV degradation, the bending strength of the 5 and 10 times dipped glass showed a decline compared to the previous state without degradation.

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Enstitümüz Kimya Mühendisliği Anabilim Dalı ortak lisansüstü programı aşağıda listelenen öğrencilerinin tez danışmanlığı Üniversitemiz Öğretim Üyesi Dr. Öğr. Üyesi Alev AKPINAR BORAZAN tarafından yürütülmektedir. Öğrencilere ait tez bilgileri aşağıda belirtilmiştir.

Bilgilerinizi ve gereğini rica ederim.

e-imzalıdır
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