

Gloss and Degradation of Hydroxyl Polyacrylic Resin/Hexamethylene-1,6-diisocyanate Coatings Under Ultraviolet and Natural-Exposure Aging

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ABSTRACT: Reactive coatings of hydroxyl polyacrylic resin (HPAR) with hexamethylene-1,6-diisocyanate were carried out under accelerated 313-nm ultraviolet (UV) aging for 2000 h and under natural exposure in Lhasa, Tibet, for 24 months. With UV irradiation and exposure time, the gloss changes in coatings with HPAR containing 3.0% or less hydroxyl groups decreased exponentially, whereas the gloss decay of coatings with HPAR containing over 4.5% hydroxyl groups decreased linearly. During 254-nm UV aging, the gloss changes in coatings with HPAR containing 1.4% or less hydroxyl groups decreased as a Gaussian function. The weather resistance of a coating was correlated to the HPAR, UV irradiation, temperature, and humidity. Scanning electron microscopy indicated that

there were degradation reactions and that some substance was lost in the matrix polymer during accelerated UV aging; then, uneven surfaces appeared and caused decreased gloss. Accelerated UV aging was faster than natural-exposure aging, and the aging velocity of 254-nm UV was 3–5 times faster than that of 313-nm UV. Through the changes in the gloss, the aging tolerance of a coating could be monitored, and its aging resistance could also be predicted. The dynamic mechanical thermal analysis results showed that the coatings had good properties. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 104: 1271–1278, 2007

Key words: ageing; coatings; degradation; films; gloss; irradiation

INTRODUCTION

Polymer molecular chains in a coating start to change both physically and chemically under exposure in nature because there are ultraviolet (UV) rays, temperature changes, humidity, oxygen, ozone, and so forth in the atmosphere. UV, temperature, and humidity are the three main factors in nature that really cause coatings to age. UV and temperature stimulate the aging reaction of a coating film, whereas humidity accelerates the degradation, weakening or breaking the polymer molecular chain structure. Even though UV light with a wavelength of 295–400 nm is about 4–7% of the total energy of sun rays in nature, its damage to a coating film is still severe. Such damage can be linked to a loss of gloss, color change, surface cracking, loss of mechanical properties, and so forth. The mechanism of gloss loss and color change is dependent on the different coating materials.¹ A loss of gloss and color fading are major defects in a coating.² The speed of gloss loss under natural exposure or accelerated UV aging reflects the

resistance to aging.³ During aging, *in situ* measurements of the mechanical properties of coating films are impossible because they would break the coating films. On the other hand, measuring the gloss would have no damaging effect on coating films. Through the change in the gloss, the aging tolerance of coatings in such an environment could be monitored.

The intensity of direct sun rays varies with the time, latitude, and altitude. Lhasa, Tibet (in China), is located at a low latitude but at a high sea level. The UV irradiation intensity from sun rays in this area is much stronger than in other areas, so the study of coating aging in this area would be more representative of the Qing-Zang high plateau in China.

The objective of this research was to examine the gloss of reactive coatings of hydroxyl polyacrylic resin (HPAR) with hexamethylene-1,6-diisocyanate (HDI) trimer (N3390) under accelerated UV aging and natural-exposure aging (NEA) in Lhasa and to predict the aging resistance of the coatings.

EXPERIMENTAL

Materials

S1, S2, and S3 were three kinds of HPARs prepared by the copolymerization^{4,5} of methacrylic acid, methyl methacrylate, butyl acrylate, 2-hydroxyethyl acrylate, and 2-hydroxypropyl acrylate. A fourth

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TABLE I
Properties of HPAR

	HPAR			
	S1	S2	S3	1903
NV (%)	50 ± 2	50 ± 2	50 ± 2	75 ± 1
OH (%)	2.1	3.0	1.4	4.5
T_g (°C)	30.8	22.5	30.0	10.5
M_n (10 ³ g/mol) ^a	9.09	3.08	21.0	No test
M_w (10 ⁴ g/mol) ^b	1.69	6.61	2.74 × 10 ¹¹	No test
Molecular weight distribution ^c	1.86	21.5	1.30 × 10 ¹¹	No test

^a Number-average molecular weight.

^b Weight-average molecular weight.

^c M_w/M_n .

HPAR [Setalus 1903BA-75 (1903)] was supplied by Akzo Nobel Resin Co. (Bergen O_p Zoom, The Netherlands). HPARs were dissolved in butyl acetate. The —OH functional group content (OH), based on the nonvolatile content (NV) in HPAR, the glass-transition temperature (T_g), and other properties are listed in Table I. NV of Desmodur N3390 BA/SN (N3390, Shanghai Co. Ltd., Shanghai, China) was 90 ± 1%, and the —NCO functional group content (NCO) was 19.5 ± 0.3%, as supplied by Bayer Co. The average particle size of the rutile-type titanium pigment (R-706, Taoyuan, Taiwan) was 0.26 μm, as supplied by DuPont Co.

Preparation of the coatings

These coatings were composed of two components: the —OH component and the —NCO component (N3390). HPAR could directly be used as the —OH component when the coatings were prepared as varnish films. R-760 was added to HPAR to make a pigment/matrix polymer ratio of 30%, and then it was ground to ≤6 μm, so that it would be the white —OH component when the coating was prepared as a white film. On the basis of the molar ratio (NCO/OH) of —NCO to —OH (0.8 : 1, 1 : 1, or 1.2 : 1), N3390 was added to HPAR or the white —OH component and then stirred thoroughly. The coating film was prepared according to the national standard of China (GB/T 1727-1992).

The coating film samples, 35–45 μm thick, for accelerated UV aging and NEA were all coated on polished iron plates (length × width × thickness = 150 × 70 × 1.5 mm³), and the film sample for dynamic mechanical thermal analysis (DMTA) was the polymer coating film.

Gloss measurements

According to the national standard of China (GB/T 9754-1988, which is equivalent to ISO 2813-1978), the gloss value of the film was measured with a QZX-60

(Tianjing, China) 60° incident angle specular gloss tester manufactured by Tianjing Instrumental Co.

Accelerated 313-nm UV aging (313-UV)

313-UV was achieved with a QUV/Se (Cleveland, OH) accelerated weathering tester manufactured by US Q-Panel Co., in which fluorescent UV-condensation-type lamps supplied the whole UV spectrum for irradiation and the central wavelength was 313 nm. The UV radiance was 0.60 W/m², and the testing strictly followed the national standard of China (GB/T14522-1993, which is equivalent to ISO 4892-1982). During the test, under 60°C the sample was constantly UV-irradiated for 4 h; then, there was no UV irradiation, but it was showered with 50°C water for 4 h. The gloss value was recorded periodically.

Accelerated 254-nm UV aging (254-UV)

254-UV was achieved by the placement of the sample under a fluorescent UV lamp that supplied the whole UV spectrum for irradiation; the central wavelength was 254 nm, the UV radiance was 0.60 W/m², and the other conditions were the same as those of 313-UV. The gloss value was recorded periodically.

Scanning electron microscopy (SEM) characterization

SEM characterization of the sample film was carried out with a KYKY-1000B (Zhongke Keyi Technology Development Co. Ltd., Beijing, China) scanning electron microscope made in China. All samples were examined at 25.0 kV after gold was sprayed to avoid electrostatic charging and poor image resolution.

DMTA testing

DMTA (Piscataway, NJ) of the sample was achieved with a Rheometric Scientific RSI (IV) DMTA tester

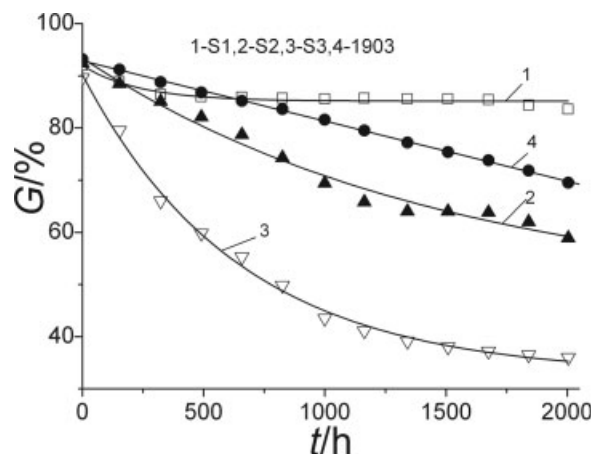


Figure 1 Gloss (G) of white films as a function of the 313-UV irradiation time (t).

made in the United States; it was heated from -70 to 250°C at a heating rate of $2^{\circ}\text{C}/\text{min}$, and N_2 was the protective gas.

RESULTS AND DISCUSSION

313-UV

The gloss was measured as a percentage. It was the ratio of the intensity of the reflex of the sample film over that of a standard plate. It was read with the 60° incident angle specular gloss tester, which is most commonly used in industry and academia. The change in the gloss was very small in the earlier beginning of 313-UV; however, the change in the gloss became large as the UV-irradiation time increased (Fig. 1).

The experimental results showed that the degradation of the coating film occurred on the surface. However, after a short induced period, spots and micropatterns appeared on the surface of the coating film, and then some cracks and minute holes appeared, which caused the loss of gloss. This was similar to the findings of Wood.² This might have been the result of oxygen combining with carbons in the matrix polymer to form some oxidized compounds in the coating films.^{6,7} These chemicals lost or formed some micropatterns on the coating surface and so caused a loss of gloss.

The gloss change of a white film under 313-UV was found to be dependent on HPAR. When OH was less than 3.0%, the changes in the gloss of the coatings of the S1, S2, and S3 HPARs reacting with N3390 were exponential:

$$G = Ae^{-\frac{t}{B}} + G_0 \quad (1)$$

where G is the gloss (%); t is the UV irradiation time (h) within the range of $0 \leq t \leq 2004$ h; and A , B , and

G_0 are constants that are dependent on HPAR. For the S1 white film, $A = 7.09$, $B = 299$, and $G_0 = 84.7$. For the S2 white film, $A = 46.3$, $B = 1495$, and $G_0 = 47.1$.

The film surface of the S3 white coating started to form minute patterns and some microblisters after 313-UV for 825 h. For the S3 white film, $A = 57.6$, $B = 649$, and $G_0 = 32.6$.

For the 1903 white film, the gloss decreased to 81.6% from 93.2% after 313-UV for 1000 h and continued to decrease to 69.5% for 2000 h because OH of 1903 was 4.5% and it needed much more N3390 to react with the $-\text{OH}$ groups in 1903. The change in the gloss was linear:

$$G = 92.9 - 0.0115t \quad (2)$$

where G is the gloss (%) and t is the UV irradiation time (h) within the range of $0 \leq t \leq 2004$ h.

According to Figure 1, during the whole 313-UV, the gloss reduction for the S1 white film was less than those of S2 and S3. The gloss reduction for the S1 white film was also less than that of 1903 after 313-UV for over 550 h. Because the molecular weight distribution of S1 was at least 1.86, it was possible to form a well-proportioned crosslinked product, a white film. Therefore, the S1 white film had the best resistance to 313-UV.

254-UV

The gloss of various white coatings decreased exponentially under 254-UV (Fig. 2). The curves were fitted with eq. (1); t was the UV irradiation time (h) within the range of $0 \leq t \leq 2000$ h.

For the S1 white coating, $A = 77.1$, $B = 278$, and $G_0 = 20.4$; for the S2 white coating, $A = 69.2$, $B = 529$, and $G_0 = 26.0$; and for the 1903 white coating, $A = 94.4$, $B = 824$, and $G_0 = 5.75$.

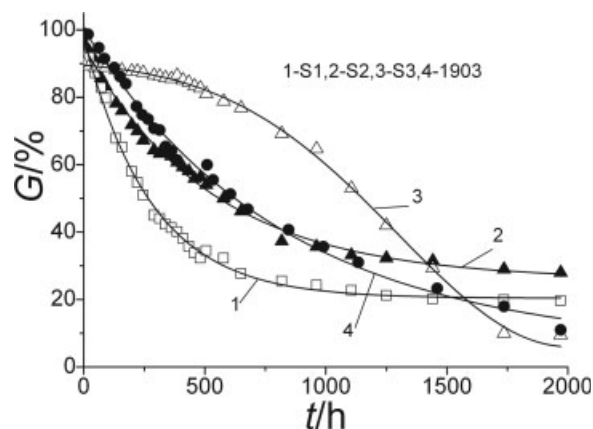


Figure 2 Gloss (G) of white films as a function of the 254-UV irradiation time (t).

Because the energy of 254-nm UV was higher than that of 313-nm UV, 254-UV stimulated a quicker photooxidation degradation reaction with the matrix polymer molecule than 313-UV. On the other hand, there were fewer hydroxyl groups (OH = 1.4%) in S3, which reacted with a smaller amount of N3390; the properties of the S3 white film were mostly attributed to those of S3. Thus, the change in the gloss of the S3 white coating under 254-UV was Gauss-shaped:

$$G = G_0 - He^{-2\left(\frac{t-t_c}{W}\right)^2} \quad (3)$$

where G is the gloss(%) and G_0 , W , t_c , and H are constants ($G_0 = 91.0$, $W = 1410$, $t_c = 2002$, $H = 85.0$).

254-UV of coatings with different NCO/OH ratios

The gloss of coating films based on different NCO/OH ratios was not identical. For example, the gloss of the S1 white film increased with an increase in NCO/OH. Under 254-UV, the gloss decay of the S1 white film fit an exponential function of the irradiation time, such as eq. (1), within the range of $0 \leq t \leq 2000$ h. When NCO/OH = 0.8 : 1, $A = 79.4$, $B = 281$, and $G_0 = 18.0$; when NCO/OH = 1 : 1, $A = 76.3$, $B = 283$, and $G_0 = 20.1$; and when NCO/OH = 1.2 : 1, $A = 75.8$, $B = 275$, and $G_0 = 22.6$.

However, the gloss of the S3 white film was different from that of the S1 white film (Fig. 3). The gloss of the S3 white film was best at NCO/OH = 1 : 1, whereas the gloss was lowest at NCO/OH = 1.2 : 1. For the S3 white films, the gloss decay fit a Gaussian function of the irradiation time, such as eq. (3), within the range of $0 \leq t \leq 2000$ h. When NCO/OH = 0.8 : 1, $G_0 = 92.0$, $W = 1589$, $t_c = 2108$, and $H = 71.9$; when NCO/OH = 1 : 1, $G_0 = 89.9$, $W = 1280$, $t_c = 2008$, and $H = 83.1$; and when NCO/OH = 1.2 : 1, $G_0 = 91.7$, $W = 1347$, $t_c = 1876$, and $H = 82.3$.

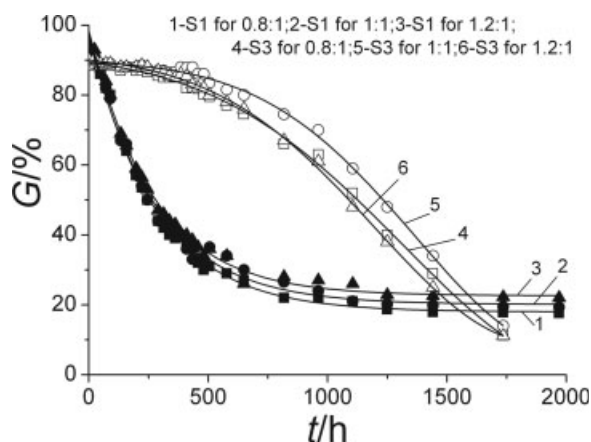


Figure 3 Gloss (G) of white films with different NCO/OH ratios as a function of the 254-UV irradiation time (t).

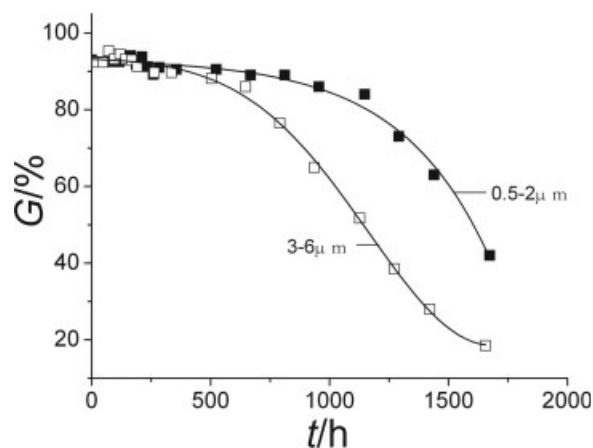


Figure 4 Gloss (G) of an S3 white film with different particle sizes of R-706 as a function of the 254-UV irradiation time (t).

254-UV with R-706 samples of different particle sizes

The changes in the gloss of the white coatings under 254-UV with R-706 of different particle sizes are shown in Figure 4. When the average particle size of R-706 in the white —OH component was 0.5–2 μm , the change in the gloss was fitted with eq. (4):

$$G = 93.2 - 0.606e^{\frac{t}{376}} \quad (4)$$

where G is the gloss (%) and t is the UV irradiation time (h) within the range of $0 \leq t \leq 1700$ h.

However, when the average particle size of R-706 in the white —OH component was 3–6 μm , the change in the gloss was Gauss-shaped and followed eq. (3) within the range of $0 \leq t \leq 1700$ h: $G_0 = 94.2$, $W = 1037$, $t_c = 1682$, and $H = 71.1$.

Therefore, the dispersion of the pigment into the resins was critical to the coating gloss. The smaller the particle size was, the better the weather resistance was and the better the gloss retention was.

NEA in Lhasa

The intensity of UV in Lhasa is much stronger than that of other areas. In Lhasa, UV rays from the sun have a characteristic distribution⁸ of a parabola, with one peak from the end of June to the beginning of August. UV radiance increases from January to July and decreases from August to December. At noon, the maximum UV radiance is in the range of 368–510 mW/m^2 , and the minimum is 22.2 mW/m^2 . The average daily UV radiance is 37–50 mW/m^2 . In the Qing-Zang plateau in China, the total intensity of sun rays a year is 695.18 kJ/cm^2 . Because the annual average temperature is around -5.2 to $+7.8^\circ\text{C}$ and the maximum difference in the atmospheric temperature between day and night is large (-45.2

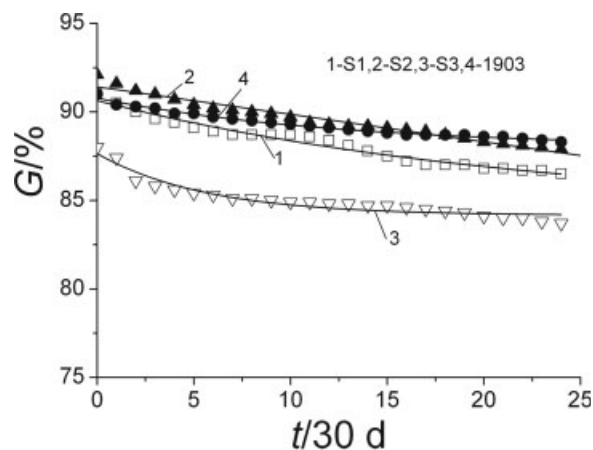


Figure 5 Gloss (G) of white films as a function of the natural-exposure time (t) in Lhasa.

to $+35.5^{\circ}\text{C}$), whereas the annual average rain precipitation is 41.8–468.1 mm and the relative humidity is 32–57%, it has been concluded that the aging velocity of a coating in such an environment is much faster than that in plain and coastal areas.

The change in the gloss of white films under NEA in Lhasa is shown in Figure 5. After 24 months of NEA in Lhasa, the gloss decreased by only 2.3–3.7%. This kind of coating was found to be very valuable for use in this snow-covered high plateau.

After mathematical fitting, the changes in the gloss of white films with different HPARs such as S1, S2, and S3 under NEA in Lhasa were developed and fitted to eq. (1), in which t was the natural-exposure time (30 days; $t \leq 24$). For the S1 white coating, $A = 6.33$, $B = 22.5$, and $G_0 = 84.3$; for the S2 white coating, $A = 2.78$, $B = 12.6$, and $G_0 = 88.0$; and for the S3 white coating, $A = 3.50$, $B = 5.66$, and $G_0 = 84.1$.

The gloss of the 1903 white coating decreased to 89.3% from 91.0% under NEA in Lhasa for 8 months and continued to decrease to 89.1% after 12 months

of exposure. The change in the gloss was linear with the exposure time:

$$G = 91.4 - 0.155t \quad (5)$$

where G is the gloss (%) and t is the natural-exposure time (30 days; $t \leq 24$).

Although these natural conditions in the Qing-Zang plateau are similar to those of accelerated UV aging, the UV radiance from direct sun rays in Lhasa is lower than that of 313-UV or 254-UV, and both the temperature and humidity in Lhasa are lower than those in 313-UV and 254-UV. On the other hand, OH in the S1, S2, and S3 HPARs was no greater than 3.0%; compared with 1903, they all reacted with a relatively small amount of N3390. However, OH in the 1903 HPAR was 4.5%, and 1903 reacted with a greater amount of N3390. Consequently, under NEA in Lhasa for 24 months, the changes in the gloss of the S1, S2, and S3 white coatings followed an exponentially shaped curve, whereas the changes in the gloss of the 1903 white film were linear.

Comparison of accelerated UV aging with NEA

The gloss changes for the S1, S2, S3, and 1903 white films under 254-UV, 313-UV, and NEA in Lhasa are compared in Table II. Because the UV radiance, temperature, and humidity in Lhasa are all lower than those in 254-UV and 313-UV, UV-, temperature-, and water-accelerating degradation to weaken or break polymer molecular chains under NEA in Lhasa is weaker than that under 254-UV and 313-UV. If natural-exposure damage to these coatings reached that under 313-UV, it would take a very exposure long time in Lhasa. For example, it would take 8–12 months of NEA in Lhasa before it had a measurable loss of gloss.⁹ Table II shows that the coating film surface would start to lose its gloss after a long time under NEA in Lhasa. This results mainly from the corrosion, degradation,

TABLE II
Comparison of Accelerated UV Aging with NEA in Lhasa

White film of HPAR	NEA in Lhasa as the reference				313-UV as the reference			
	313-UV	NEA		254-UV	313-UV	NEA	254-UV	
	Time (h)	Gloss (%)	Time (30 days)	Time (h)	Gloss (%)	Time (h)	Time (30 days)	Time (h)
S1	171	88.7	8	31	86.7	500	22	42
S1	194	88.4	12	34	85.6	1000	36	47
S1	410	86.5	24	35	83.6	2000	Very long	55
S2	114	90.0	8	36	81.1	500	Very long	121
S2	139	89.2	12	41	69.4	1000	Very long	247
S2	174	88.3	24	47	58.9	2000	Very long	393
S3	60	85.1	8	347	59.4	500	Very long	1010
S3	64	84.8	12	374	43.5	1000	Very long	1241
S3	78	83.7	24	389	36.0	2000	Very long	1344
1903	313	89.3	8	95	86.7	500	30	127
1903	330	89.1	12	101	81.6	1000	63	180
1903	435	87.9	24	103	69.5	2000	141	323

TABLE III
DMTA Results

HPAR	White film				Varnish film			
	E'_{\max} (10^9 Pa)	E''_{\max} (10^8 Pa)	T_g ($^{\circ}\text{C}$)	ΔT_g ($^{\circ}\text{C}$)	E'_{\max} (10^9 Pa)	E''_{\max} (10^8 Pa)	T_g ($^{\circ}\text{C}$)	ΔT_g ($^{\circ}\text{C}$)
S1	3.90	2.99	55.9	25.1	3.09	2.08	51.0	20.2
S2	4.88	3.90	44.1	21.6	3.09	2.50	43.1	20.6
S3	4.68	4.00	41.3	11.3	3.26	2.70	44.0	14.0
1903	3.98	2.80	46.9	36.4	3.80	2.62	42.1	31.6

photon oxidation, and evaporation of small molecules. Accelerated UV aging is faster than NEA because of the higher UV energy and radiance, temperature, and humidity. The aging velocity of 254-UV is 3–5 times faster than that of 313-UV.

DMTA

According to DMTA, T_g of varnish and white films of S1, S2, S3, and 1903 reacting with N3390 was

defined as the temperature at which $\tan \delta$ was maximum. The storage modulus, loss modulus, and T_g values of the white films were higher than those of the varnish ones. They all had a higher modulus in the glassy state. The results of DMTA indicated that they were in the glassy state at the atmospheric temperature; the maximum storage modulus (E'_{\max}) and maximum loss modulus (E''_{\max}) were over 10^9 and 10^8 Pa (Table III), respectively. When the temperature was in the range of 65–250 $^{\circ}\text{C}$, the polymers in

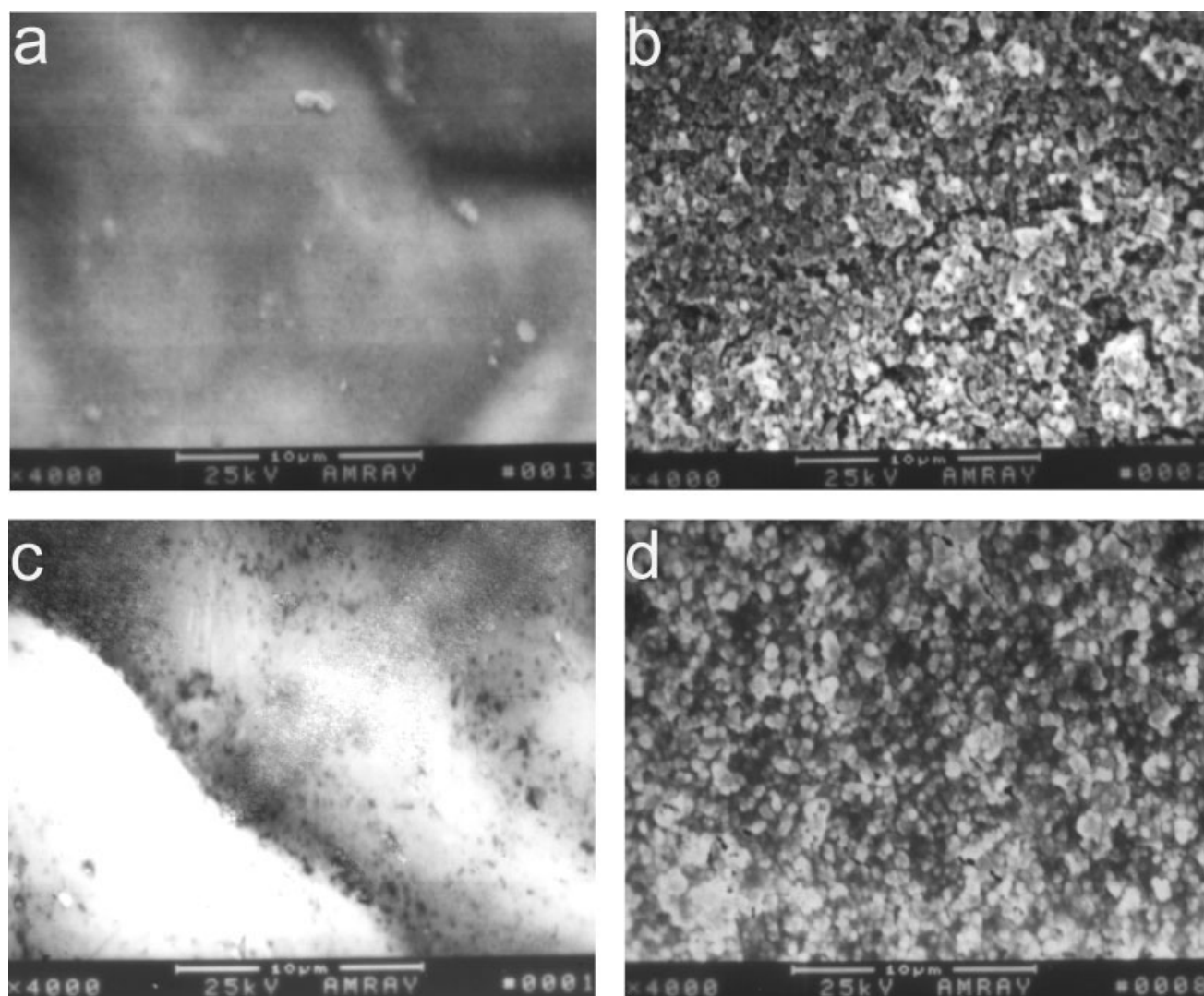


Figure 6 SEM images of white-film surfaces (a,c,e,g) before and (b,d,f,h) after 254-UV irradiation for 2000 h: (a,b) S1, (c,d) S2, (e,f) S3, and (g,h) 1903.

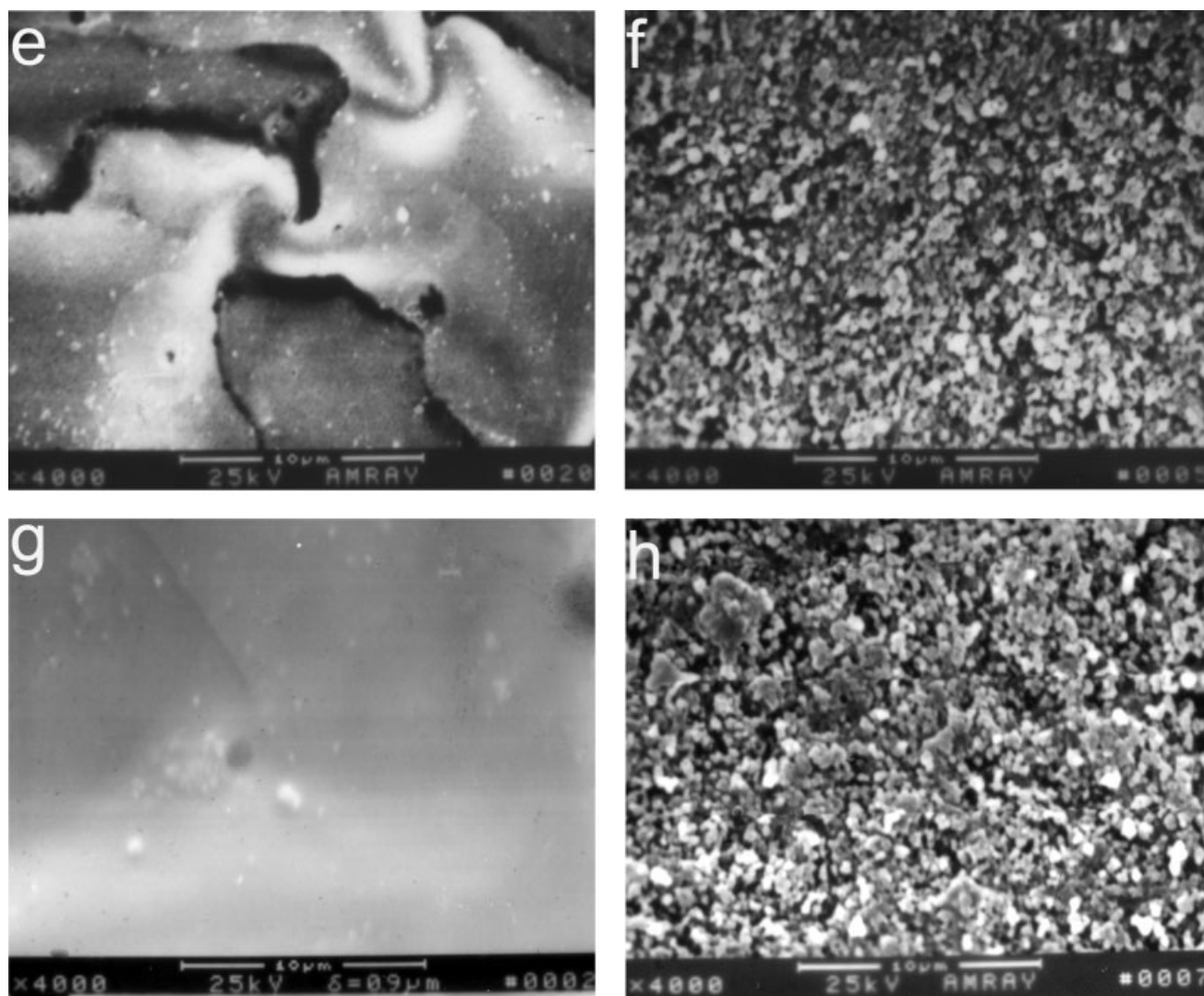


Figure 6 (Continued from the previous page)

these varnish and white films were in the rubbery elastic state; however, their storage moduli were in the range of 10^6 Pa.

If the S1, S2, S3, or 1903 HPAR was considered to be a pure phase, its T_g was apparently unmoved¹⁰ as the error of DMTA in measuring T_g was approximately $\pm 2^\circ\text{C}$. The T_g values of the S1, S2, S3, and 1903 HPARs were 30.8, 22.5, 30.0, and 10.5°C (Table I), respectively. For the S1, S2, S3, and 1903 HPARs, the T_g 's of their varnish and white films all shifted to higher temperatures. Their shifts (ΔT_g) in HPAR are shown in Table III. As a result, with an increase in OH in HPARs, the amount of N3390 in the coating increased, and ΔT_g 's of their varnish and white films also increased. Table III, however, shows that the ΔT_g values of the S3 varnish and white films were 14.0 and 11.3°C , respectively. This indicated that the property of the S3 varnish or white film was mostly contributed by the S3 HPAR.

It also indicated that the properties of the S1, S2, and 1903 varnish and white films were contributed by the S1, S2, and 1903 HPARs reacting with N3390 because their ΔT_g 's were over 20°C .

SEM analysis

SEM images of white film samples are shown in Figure 6. Before 254-UV, 2- or $6\text{-}\mu\text{m}$ R-706 was completely dispersed in the matrix polymer, and the white films were smooth, uniform, close, and consistent. Thus, it was possible to inhibit UV irradiation into the film so that the film had good resistance to UV.

However, the S3 white film had a block morphological structure [Fig. 6(e)]. Because OH of S3 was small (1.4%) and it reacted with a smaller amount of N3390, the molecular weight distribution of S3, 1.31×10^{11} (Table I), was too large. S3 had a contin-

uous phase in its coating, and the completely dispersed R-706 was not only the disperse phase, whereas the site of S3 reacted with N3390 but also equated with another disperse phase. This formed a block–block morphological structure, so water permeated the S3 white film; this promoted water absorption and caused minute patterns or blisters to form after 313-UV for 825 h. This indicated that this coating would not be good in UV aging at a higher temperature ($\sim 60^{\circ}\text{C}$) and moisture content. This was consistent with the results of 313-UV and DMTA analysis.

After 254-UV for 2000 h, the S1, S2, S3, and 1903 white films all gave similar results; on their surfaces were spots, patterns, microcracks, and microholes around the R-706 particles [Fig. 6(b,d,f,h)]. The R-706 particles were almost exposed.

We proposed there was a possible degradation mechanism for the coatings under UV and NEA. During accelerated UV aging and NEA, there were small-molecule substances to form and lose, there were some degradation reactions of the matrix polymer in the coating, or there were still some soluble degradation products or some soluble components in the coating, which caused osmotic pressure effects on the coatings to promote water absorption. On the other hand, the matrix polymer might be in the rubbery elastic state at a higher temperature ($\sim 60^{\circ}\text{C}$), and more water was absorbed into the film to form some blisters for a longer wet period under accelerated UV aging and NEA. As a result, the surfaces of the coatings were not consistent or smooth; some small cracks appeared, and the filler (R-760) particles were almost exposed. Finally, the gloss and mechanical properties decreased. UV damage to the coating films was the most severe. Simultaneously, temperature stimulated the aging reaction of the coating

films, whereas water accelerated the degradation reaction to weaken or break the polymer molecular chain structure.

CONCLUSIONS

Coatings aged faster under accelerated UV aging than NEA. UV played a key role in the aging process. Gloss measurements *in situ* were used to monitor the aging tolerance of coatings during the aging process. The aging speed was dependent on the matrix polymer of the coatings, the temperature, and the humidity. The aging speed in 254-UV was 3–5 times faster than that in 313-UV. Therefore, the simulation of accelerated aging could be conducted with UV with a shorter wavelength to save experimental time and to predict the aging resistance of coatings.

This kind of coating has good UV resistance. The weather resistance is also over 2000 h under 313-UV. It is suited for protective coatings for areas of strong UV irradiation.

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