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THE DARK SIDE OF BRIGHT METAL COATINGS

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Abstract

The surface quality of light reflective metal films on polymer substrates were experimentally examined and evaluated. The study included light optical hotstage microscopy of in-situ samples and scanning electron microscopy. Conditions for producing aluminum metal films with desirable light reflecting properties are relatively demanding. The film formation is a complex series of events which include nucleation, coalescence and crystal growth.

Surface flaws, debris or other imperfections are readily visualized with the aid of the optical or the electron microscope. Although complex methods have been developed for the examination of small surface areas with fine-featured metallic films, there was little incentive to adopt the techniques for large samples. This situation prevailed because the simple microscopic evaluations were sufficient to reveal unacceptable qualities.

Hotstage microscopy was decisive in understanding the light reflectivity of the metalized layer and the substrate's influence over the deposited film. Prior to the hot stage examination, only the thin section based knowledge supported the film evaluations. There was always the disturbing possibility that some mechanically or chemically introduced artifacts were contributing to the observed surface irregularities and distortions.

The hotstage experiments proved that the observed surface corrugations are the result of complex mechanisms which may depend on several factors. These contributing factors include the combined results or the individual interaction between substrate, base coat, residual stress and temperature. Surface corrugations reproduced with the hotstage yielded valuable informations. In some instances, the experiments, pinpointed the underlying cause and in others it indicated the role of the variables.

KEY WORDS: bright metal coatings, hotstage microscopy, interference colors, reflectors, injection molded polycarbonate, residual stress, surface corrugations, optical microscopy.

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Introduction

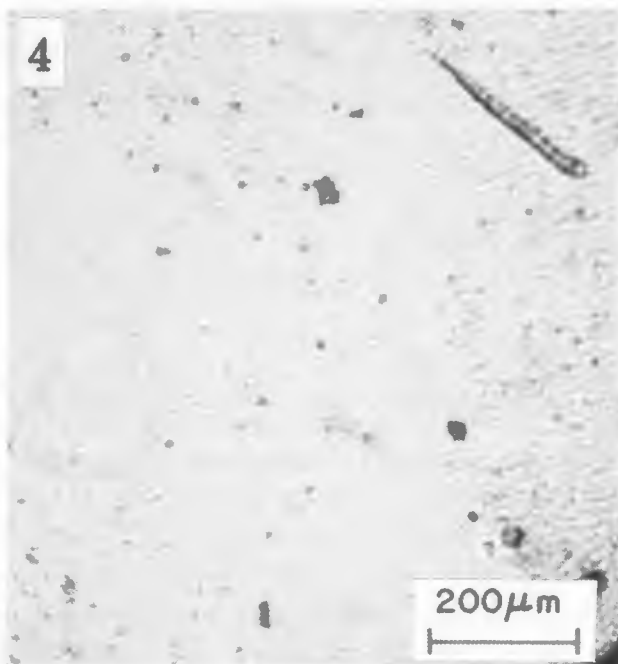
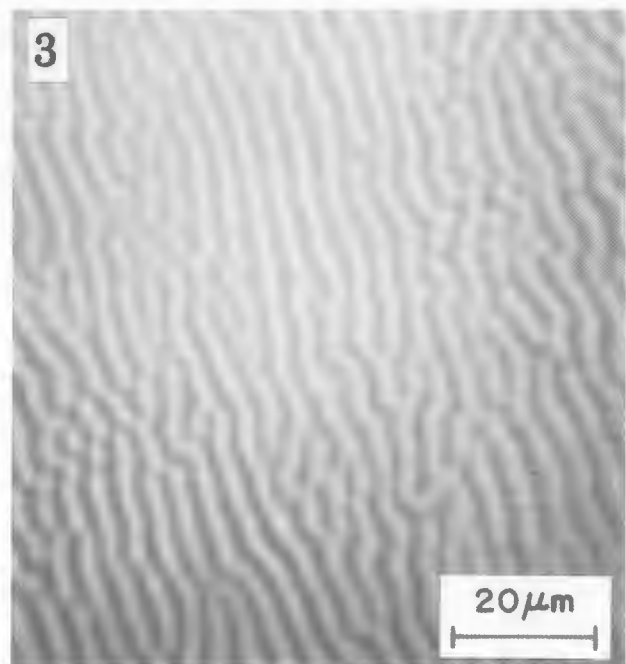
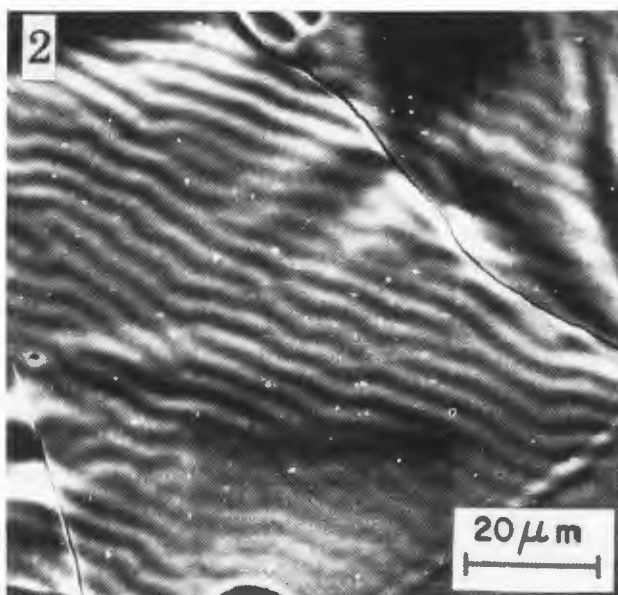
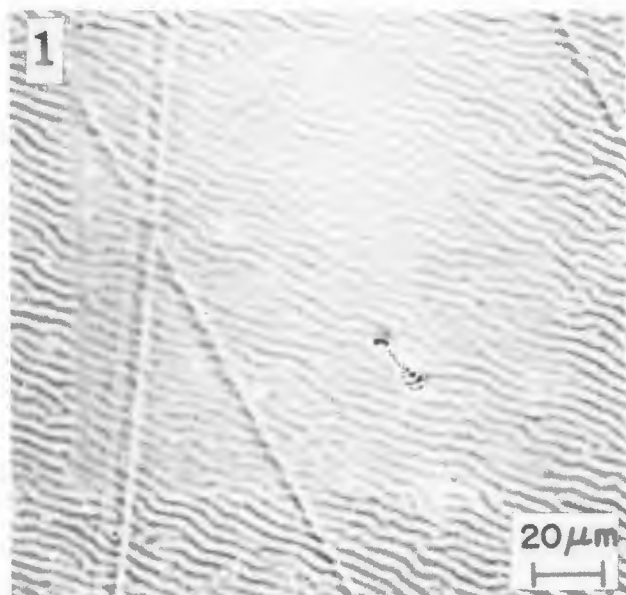
Reflectors are made from a polycarbonate shell which is coated first with a polymer base coat, then with an aluminized layer and finally with a transparent lacquer layer to protect the surface. Optical hotstage experiments indicate that the base coat, located under the aluminized layer, is mainly responsible for the wrinkled surface which produces the interference colors reflected from the metallized layer. In addition, residual stresses in the injection-molded polycarbonate reflector shell play an important role. Separate hotstage experiments were used to study the defect forming role of the base coat and the polycarbonate shell residual stresses. These experiments could not fully separate all the factors. But, with various time and temperature exposures, the role of the base coat and the residual stress were clearly demonstrated.

Surface changes triggered by the base coat are especially noticeable at higher temperatures owing to the typically coarse surface corrugations. The curing temperature of the top lacquer layer (approximately 125°C) was found to be incompatible with the base coat. In addition, the stressed surface of the injection-molded polycarbonate shell relaxed at the lacquer curing temperature and produced fine-structured surface defects. These surface anomalies, at temperatures above the lacquer curing temperature, remained fine when no base-coat was present indicating that stresses and not the base coat caused the defects.

Materials and Methods

Sample Description

Three different reflectors were examined. The aluminized surface of one reflector was defective⁴ and produced interference colors visible to the naked eye. This defective aluminized surface is supported by a polymer base coat and the assembled reflector had been heated to cure the top lacquer layer. The aluminized surfaces of the other two reflectors were defect-free and did not show any interference colors. One of these reflectors was coated with a polymer base coat, the other was not. Both of these defect-free reflectors were lacquer coated and neither had been exposed to the lacquer curing temperature.



Figs. 1 - 3. Defective reflector surface with coarse corrugations. The uniformity of the wrinkles is noteworthy. The reflector has cured top coat, Al layer, and a base coat.

Fig. 4. Typical defect-free reflector surface. The reflector has base coat, Al layer and an uncured top coat.

Microscopic Examinations

Examination of the defective reflector surface with scanning electron microscopy (SEM) and incident light optical microscopy revealed a "diffraction grating" or line-type structure². This line-type structure is the result of very fine surface wrinkles. The wrinkles are equidistant parallel peaks and valleys which give the overall lined appearance. Figures 1 and 2

are scanning electron micrographs which show the line-type surface structure of some different areas at varied magnifications. Figure 3 shows similar corrugations using light optical microscopy. The peaks are approximately 1 to 2 microns high which explains the slight out-of-focus image in Figure 3. The peak-to-peak linear distances for several areas are uniform and average approximately 240 lines per mm.

THE DARK SIDE OF BRIGHT METAL COATINGS

The surfaces of the two defect-free reflectors were also examined and a typical surface structure is shown in Figure 4. The base-coat-supported surface has imperfections and a fairly large number of contaminating particles. These surface imperfections are not the uniformly spaced line-type structure, therefore, interference colors are not produced. However, reflectivity can still suffer due to these surface anomalies.

The aluminized layer was partially removed from a defective reflector by mechanical means to examine the base coat surface. This base coat surface has a lined or corrugated structure as shown on the left side of Figure 5. The structure appears coarse, because there is little contrast between the peaks and valleys with no aluminum highlighting. On the right side of Figure 5, the lined surface structure of the aluminized layer is visible. The "washed out" appearance of the image is due to imaging difficulties with the two different light reflections and not the result of a damaged, diminished, or changed surface. The compromise in photographic exposure was necessary to accommodate the base coat's poor light reflectivity.

Temperature Effect on Base Coat

It was assumed that corrugation of the base coat was responsible for the wrinkling of the aluminized layer. Corrugations may result from the rapid evaporation of solvents, stress, temperature or combinations of these factors. To study the defect-forming process, optical hotstage experiments were conducted. For the first experiment, samples were prepared at random from the defect-free aluminized surface of the reflector that had a base coat and an uncured top coat. The hotstage experiment started with the room-temperature image of the aluminized surface layer and continued with selective imaging during the entire heating experiment. Figure 6a illustrates the surface at room temperature. The contaminants shown were purposely included to serve as landmarks because slight sample movements are unpredictable during a lengthy hotstage experiment. These features help to identify the same areas when photographed at different magnifications if slight displacements occur. The image in Figure 6a was taken at a relatively low magnification to assure that the large field of view with numerous particles would accommodate higher magnifications or slight sample displacements.

The sample was heated at the rate of 4°C per minute from room temperature to 110°C. The subsequent heating rate was reduced to 0.5°C per minute to permit visual observation of the changes. Figures 6b, c, and d, show only minor and gradual changes of the surface. The first major change in the surface appearance is seen at 139.5 °C and is shown in Figure 6e. In the lower part of the micrograph, the initial development of the line-type structure is visible. Above this area, the micrograph gives some evidence of the development process. The line-type structure development is a gradual process. These surface changes are controlled by the base coat because the aluminized layer is unable to resist the mechanical forces⁶. The precise behavior and role of the base coat is not clear, however. The observations suggest that the base coat surface wrinkling or ridging with temperature is similar to rapid-evaporation-caused

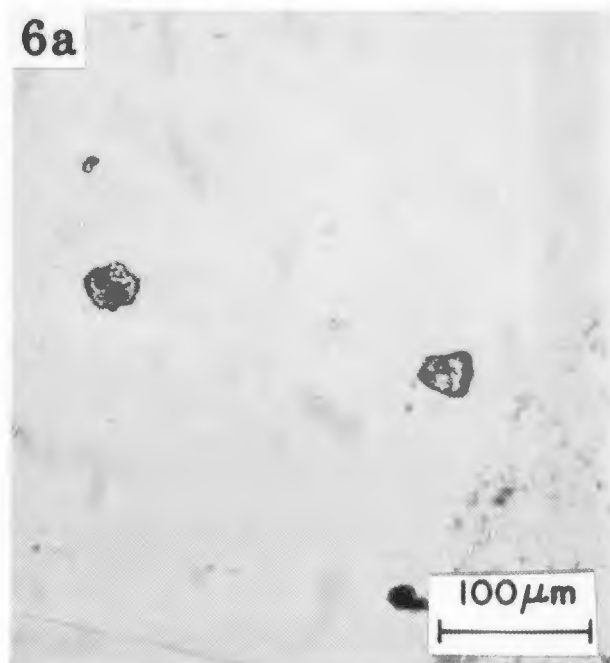
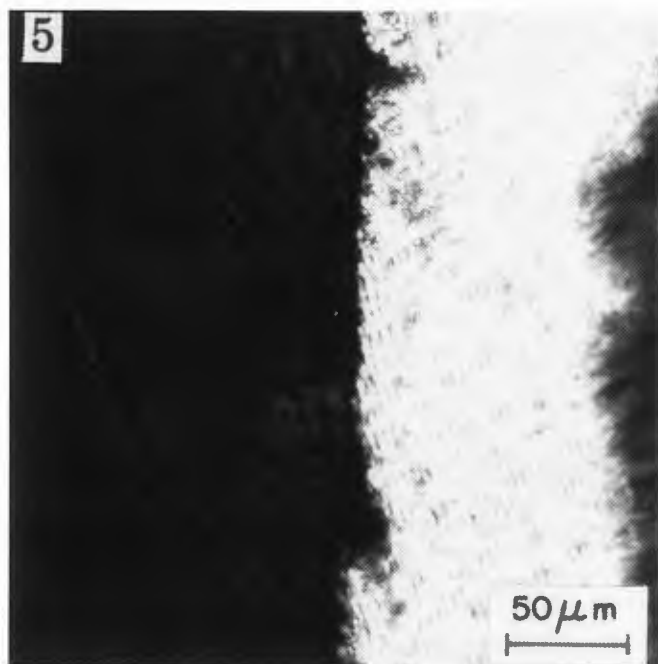
wrinkling. At 139.5 °C, and slightly above 140°C, the surface corrugations occur rapidly and they are coarse. At 142 °C the surface alteration is complete. Figures 6f and g, show the intermediate and the complete change, respectively.

Some surface changes were also observed below the polycarbonate glass transformation temperature. Therefore, association with the stressed state of the polycarbonate reflector shell, induced during the injection-molding process, could not be eliminated as a factor. As will be discussed later, not all surface changes caused by stress are visible using the optical microscope. Comparison of hotstage-produced surfaces with surface defects produced during manufacturing shows an interesting difference. Figure 7 reveals the line-type structure of the "as received" defective reflector surface. This sample has top and base coat and it was exposed to the lacquer curing temperature. The line-type structure is fine and the directional orientation indicates the mode and direction of the polymer base coat application. The base coat was smeared, not sprayed, on the surface of the polycarbonate reflector shell. Had spraying been used, the long chained polymer layer would not have formed a uniformly oriented wrinkled or "lined" surface structure. The application direction was normal to the orientation of the lines. This observation is in general agreement with the behavior of long-chain polymers and their response to temperature. Some lines, however, lack continuity and terminate after a short distance. For these, the explanation may lie with the base coat thickness or uniformity or perhaps the aluminized layer thickness³.

The hotstage experiments were successful in approximately duplicating the appearance of the manufacturing induced interference colors. Exposing an uncured sample with base and top coat in the hotstage for 2 h at temperatures between 118°C and 125°C produced refined and directionally oriented line-type structures; Figures 8a and b are typical. It is worthwhile to repeat that higher temperature produced coarse surface corrugations (Figures 6f and g), but the initial surface changes may occur at temperatures as low as 118° to 120°C.

To prove that the base coat is responsible or even associable with the observed surface defects, an additional hotstage experiment was conducted. Samples were prepared at random from a defect-free reflector with no base coat and no lacquer cure. The sample was heated at a rate of 3 °C per minute in the hotstage. Figure 9a illustrates the room temperature image of the metallized reflector surface. The surface imperfections and the contaminating particles are typical. From room temperature to 138°C the surface changes are minor and nearly undetectable as shown in Figures 9b, c, and d. The lack of photographically detectable surface changes are evidence that a short exposure to temperatures up to 138°C (the glass transition temperature of polycarbonate) does not affect the surface appearance. If significant surface stresses were present, a longer annealing time below the glass transition temperature might still affect the surface appearance.

A noticeable change at this temperature involves only the reduction in the number and size of the contaminants. The defect size reduction was caused by evaporation because no stain



or other surface changes are visible. In addition, the cover glass of the hotstage chamber received a slight deposit indicative of evaporation. In the absence of visible surface changes the experiment involving this base coat free sample was terminated at 145°C.

The absence of additional changes or of any changes similar to those seen earlier on the base coated reflector indicates that the light reflectivity of the aluminized layer is dependent on exposure of the base coat to high temperature. Thus the experiment suggests that the base coat was not compatible with the temperature used to cure the top lacquer layer.

Annealing

To investigate the effects of an extended anneal on the surface structure another sample without base coat and with an uncured top coat was tested in the hotstage. The aluminized surface of the sample was examined in the microscope before the hotstage experiment. The surface appearance (not shown) was nearly identical to the image shown in Figure 9a. Before placing the sample in the heating chamber, the chamber was preheated and stabilized at 122 ° C which is approximately the curing temperature of the lacquer layer.

With extended annealing, the surface was substantially altered and Figures 9e through 9g show the changes. After two hours of continuous anneal (Figure 9g) the surface appears remarkably similar to the manufacturing produced surface shown earlier in Figure 7. The experiment indicates that the level of changes and fine details are time and temperature dependent. It is also interesting to note the gradual development of the lines from the coarse to the refined structure as shown with Figures 9e, f, and g. The coarse surface that developed during the first 20 minutes of anneal retains the basic structural character for an additional hour, but at the end of two hours at 122°C substantial changes have occurred. This sample had no base coat and the observed surface changes were caused by time and temperature, which can be associated with the stress relaxation of the reflector polycarbonate shell, and will be discussed later.

Base Coat Curing

During hotstage experiments it was noticed that the surface corrugation of the "as received" defective reflector changed with the increase or decrease of the temperature. These changes were assumed to be associated with the curing temperature of the base coat. To test this hypotheses two additional hotstage experiments were conducted to ascertain the effect of the base coat curing temperature. Except for the controlled cooling rate, the experimental parameters of both studies were similar to those used in the earlier hotstage examinations. The heating rate was 3°C per minute and the cooling rate was approximately 5°C per minute. Samples were selected at random from reflectors that had the uncured base and top coats. For the hotstage experiments the samples were individually pre-cured for two hours using two different temperatures. One

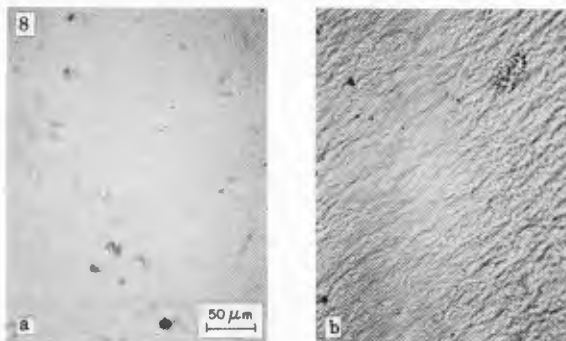
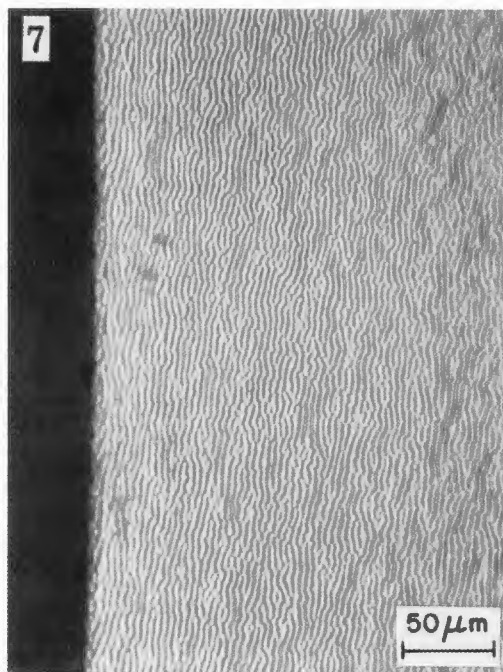


Fig. 7. Defective reflector surface with fine corrugations and cured top coat.

Fig. 8. Hostage produced surface anomalies with fine and directionally oriented corrugations. (a) at 118 C, and (b) at 125 C. Figs. 8 (a) and (b) are at same magnification, note bar in Fig. 8 (a).

sample was precured at slightly lower than 120 ° C; the other at 135 ° C. Only the lower-temperature-pre-cured sample attained the altered surface appearance during the hotstage experiments because the higher precure temperatures ended all surface changes.

Surface corrugation, caused by the lower temperature pre-cure, is shown at room temperature (Figure 10a). The image is shown at high magnification to aid a later comparison with the changed line-type structures of the surface that occurred after the sample was heated and cooled

Fig. 5. Surface appearance of base coat and the aluminized layer. The sample's top coat is cured.

Fig. 6. Experimentally produced surface changes with the optical hotstage. (a) At room temperature and low magnification; (b) at 110 C; (c) at 120 C; (d) at 135 C; (e) at 139.5 C; (f) at 140.5 C; and (g) at 142 C. Figs. 6 (b) - (g) are at same magnification; note bar in Fig. 6 (c).

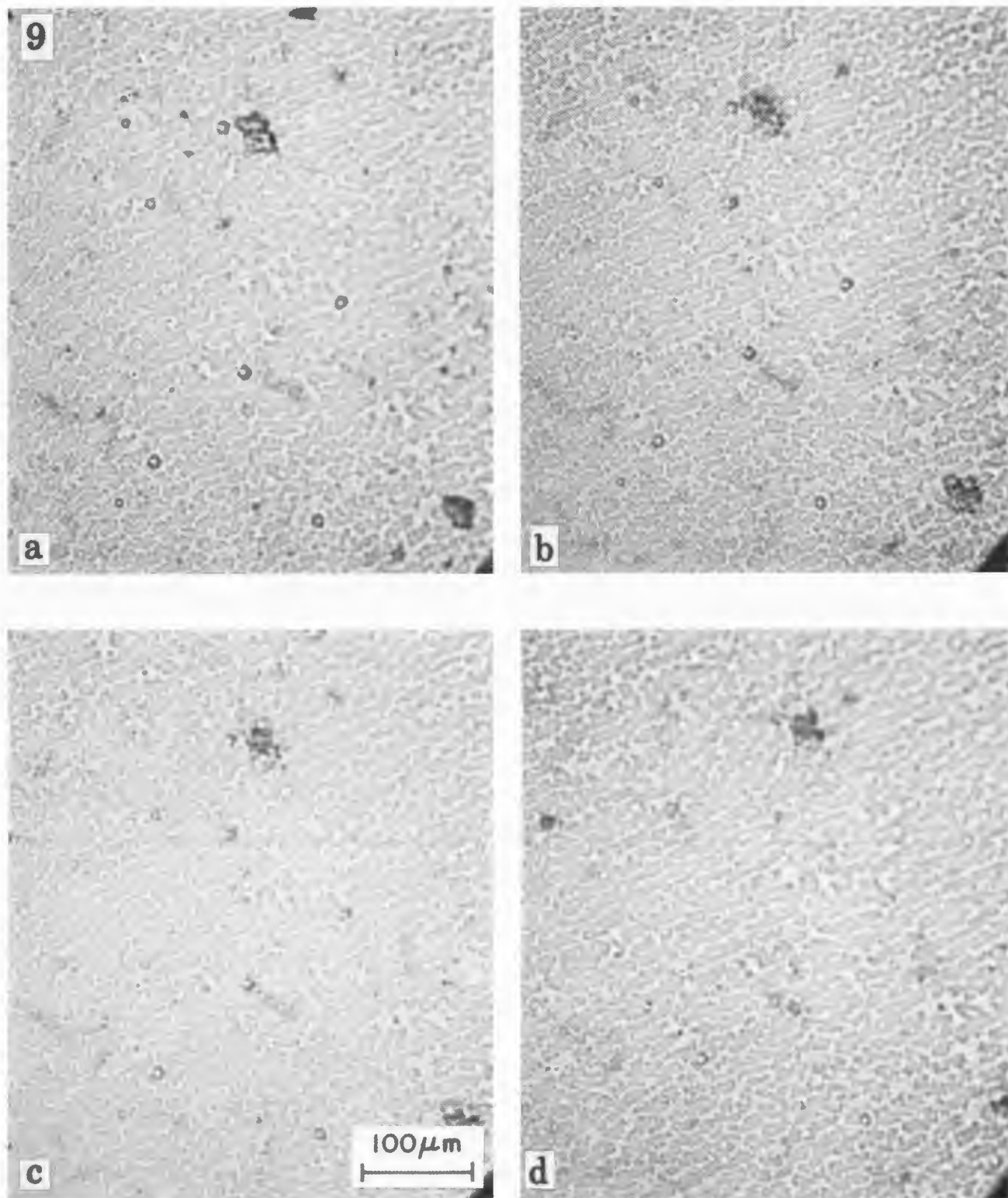
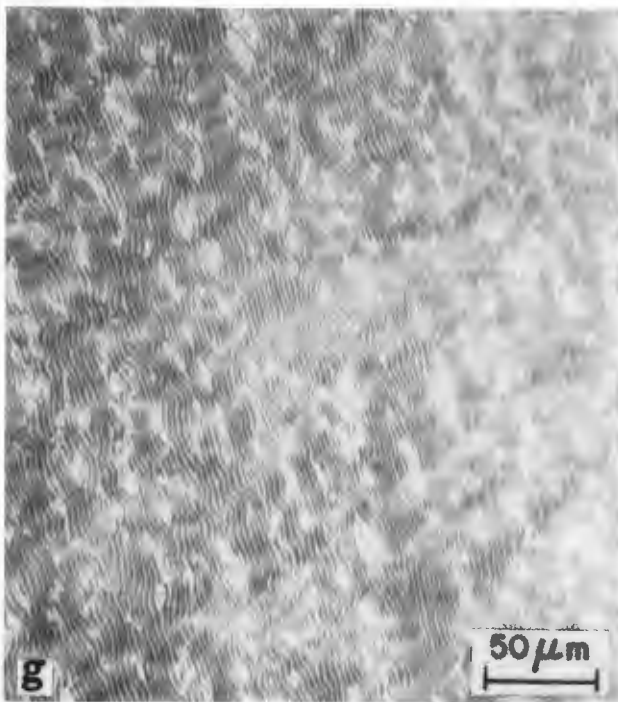
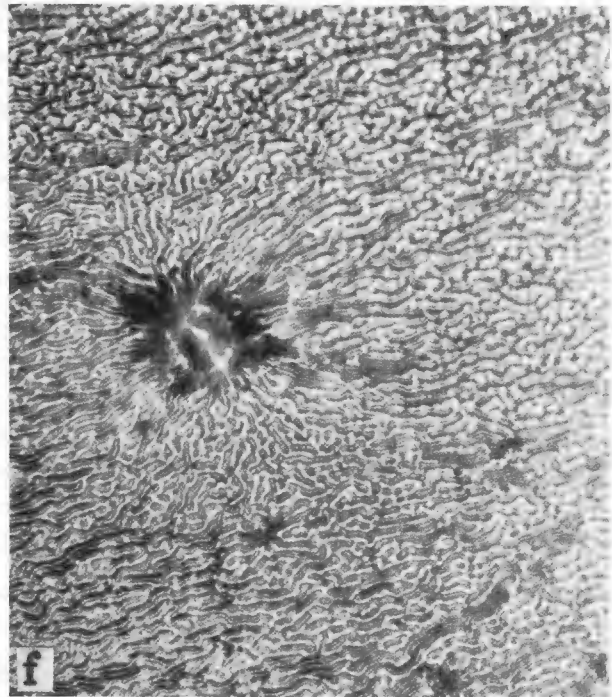
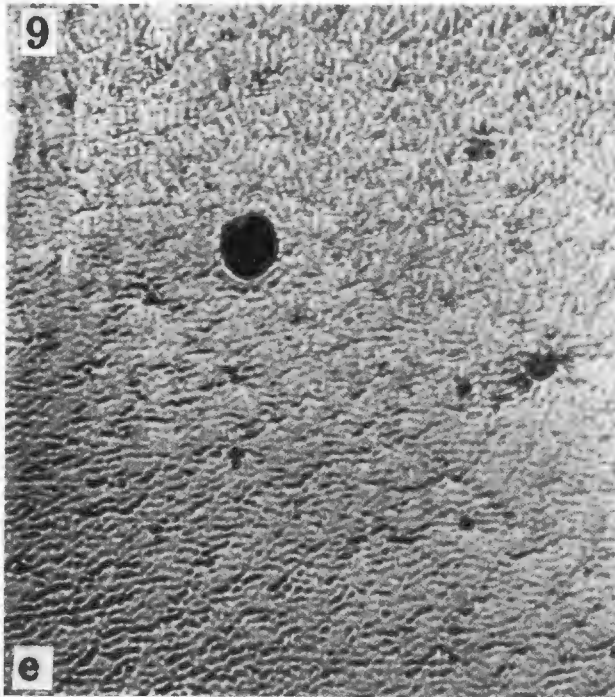


Fig. 9. Appearance of various surface defects produced with the optical hostage. The samples were uncured and without base coat. (a) at room temperature; (b) at 110 C; (c) at 120 C; (d) at 138 C; (e) after 20 minutes aging at 122 C; (f) after 1 hour aging at 122 C; and (g) after 2 hours aging at 122 C. Figs. 9 (a) - (d) are at same magnification, note bar in Fig. 9 (c); Figs. 9 (e) - (g) are at same magnification, note bar in Fig. 9 (g).



in the hotstage. With the exception of Figures 10a and 10c, one large contaminant at the bottom of all micrographs identifies the areas shown in Figures 10a - 10h. The hotstage experiments indicated that below 110°C the overall surface changes and the smoothing of the corrugations are minimal. The most noticeable changes occurred at 110 ° C or a few degrees above. Figure 10b illustrates the altered appearance. For all

practical purposes the surface rearrangements are "finalized" and there are no other appreciable changes even at higher temperature. To support this observation, Figure 10c shows the smoothed aluminized surface of an identical sample at 145°C, during a duplicate hotstage experiment; the images are almost identical.

The effect of the cooling rate on the surface structure is dramatic. Careful examination of the surface, cooled by only 10 ° C, (to 100 ° C) already demonstrates a stronger line-type structure, and in the center of Figure 10d, the emerging lines have some additional bends and extensions. The effect of further cooling is documented in Figures 10e through 10h. The continuity and shape of the individual lines appear to be "final" at 90°C (Figure 10e) but the detail visibly "matures" between 60°C and 45 ° C as shown with Figures 10g and h respectively.

During the hotstage heating and cooling experiment, the line-type structure of the surface changes from its pre-cure-produced appearance. Comparing the room-temperature image of the pre-cured surface (Figure 10a) to the images of the cooled surfaces (Figs. 10f, g, h), only minor changes are visible. The most noticeable is in the vicinity of the landmark contaminant. This indicates that after the pre-cure temperature of approximately 120 ° C, further changes have occurred in the base coat. This reasoning is reinforced by the second experiment using the 135 ° C pre-cured sample. Some microscopic surface smoothing was observed when heated to 110°C, but when the sample was cooled to 45 ° C there was no visible surface change from the corrugation produced by the 135°C pre-cure. This indicates that the base coat was fully cured and not prone to further change.

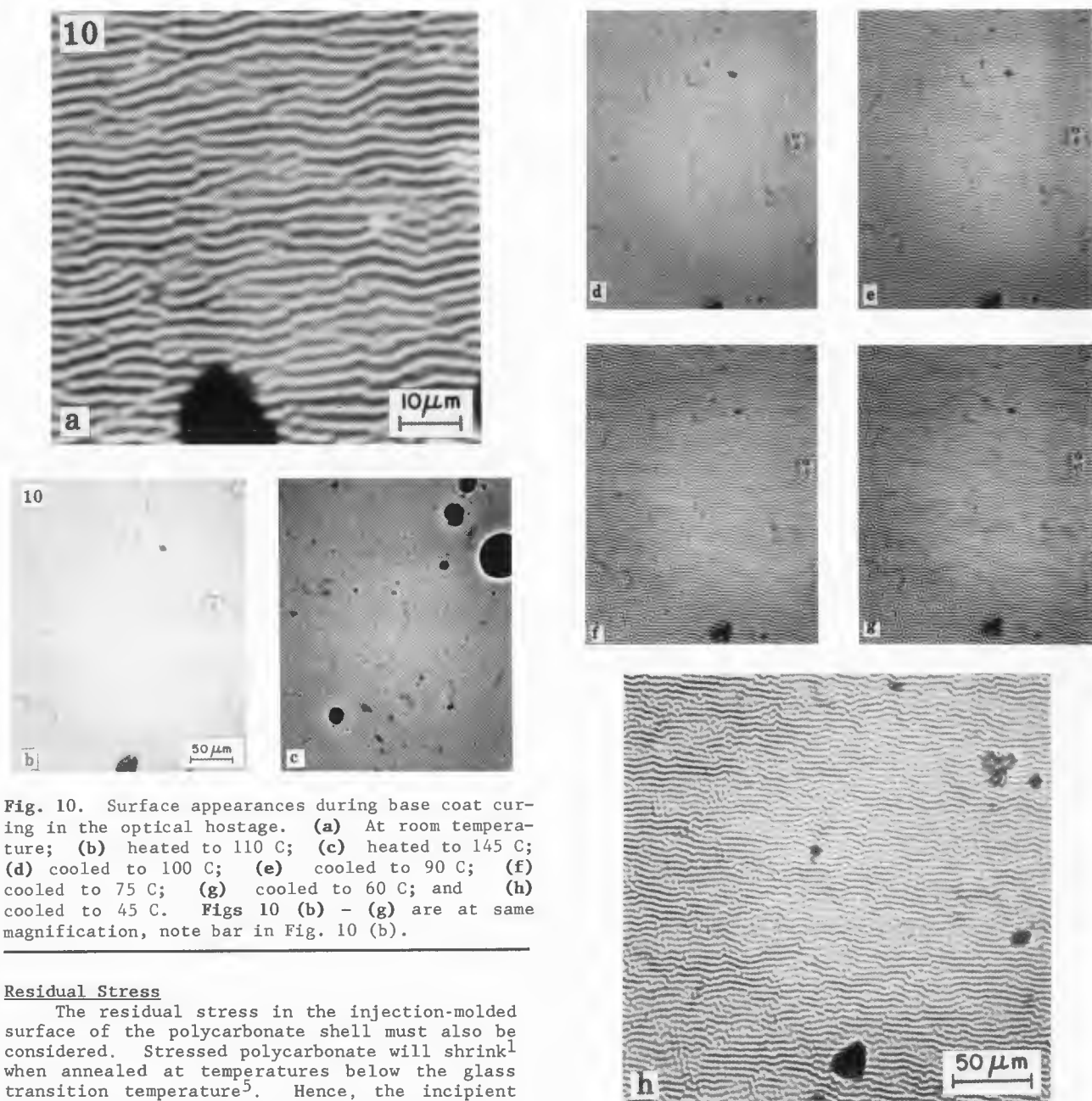


Fig. 10. Surface appearances during base coat curing in the optical hotstage. (a) At room temperature; (b) heated to 110 C; (c) heated to 145 C; (d) cooled to 100 C; (e) cooled to 90 C; (f) cooled to 75 C; (g) cooled to 60 C; and (h) cooled to 45 C. Figs 10 (b) - (g) are at same magnification, note bar in Fig. 10 (b).

Residual Stress

The residual stress in the injection-molded surface of the polycarbonate shell must also be considered. Stressed polycarbonate will shrink¹ when annealed at temperatures below the glass transition temperature⁵. Hence, the incipient wrinkling of the aluminized layer, observed at about 120°C, may have also been associated with the shrinking of the polycarbonate shell surface. To verify this suspicion two more hotstage experiments were conducted.

Samples were prepared at random from the base coat free reflector shell that had an aluminized surface and an uncured top lacquer layer. For the first experiment, the wall thickness of the reflector body shell was mechanically reduced to one third of its original thickness by a jeweler's saw. To avoid heating the sample during the sectioning the cutting was slow and water was used to cool the sample. The aluminized surface was examined before and after the wall thickness reduction with the microscope. This thickness reduction was postulated to partially relax the residual stress in the shell wall and thus reduce

the surface corrugation triggered by time and temperature. The sample's metallized surface was carefully examined for details at room-temperature and no surface changes⁷ were noted following the thickness reduction. The surface is not shown but it is similar to the image of a typical base coat free uncured sample shown in Figure 9a.

The sample was not gradually heated to the annealing temperature but rather introduced into the temperature stabilized hotstage environment preheated to the 122°C annealing temperature. The surface appearance of the sample was periodically photographed and Figures 11a through 11d show the changes. After the first hour of anneal the sample surface has mottled, the luster

decreased, and a faint line-type structure is developing. The surface changed very slowly and gradually. Only after three and four hours was a more noticeable line-type structure produced. At the end of four hours the line-type structure was still not sufficiently developed to produce interference colors. Because the corrugations were shallow and lack a well-ordered repetitive pattern, no interference colors were observed. In addition, the distance between the peaks and valleys was not uniform.

For the second experiment a full wall thickness sample was prepared from the same reflector body shell. The area selected for this sample was adjacent to the location from which the reduced wall thickness sample was obtained. The expectation was that a full wall thickness sample would show a more drastic response to aging at the 122 ° C annealing temperature; this was indeed observed. With the exception of the aging time, the other hotstage parameters (temperature, chamber stabilization, imaging) were identical to the procedure used during the preceding experiment. Figures 11e through 11h show the changes. The beginning of the surface corrugation becomes visible in the first 15 minutes of anneal (Figure 11e). After 30 minutes, the orientation and direction of the lines is clearly established with fine and continuous pattern (Figure 11f). Subsequent anneal further coarsens the corrugations and the apparent line-type structures are shown in Figures 11f, g and h. Noticeable differences took place each 15 minutes of subsequent anneals. The changes were much more pronounced than previously seen in the reduced wall thickness sample. The experiment was terminated after one hour because measurements indicated (by the peak heights and peak-to-peak distances) that a close reproduction of the interference color producing line-type structure had been developed. Since neither the reduced nor the full-wall-thickness sample had a base coat and both developed surface anomalies, the experiments present clear evidence for the role of residual stress in forming the interference color producing line-type surface structure.

Summary

The underlying mechanism and forces for the variable appearances of the surface corrugations or lines are not yet fully understood. The observed and experimentally reproduced phenomena of surface wrinkling is reminiscent of stress relaxation produced surface flaws. And some are.

The results of the hotstage experiments, that used base coat-free samples with full and reduced wall thicknesses, were directly associable with the molded-in stress of the polycarbonate substrate. On the other hand the precise role or influence of the base coat for the defect forming process has not been established. Only the temperature related observations were evaluated.

Acknowledgements

The authors wish to thank Dr. H. van Oene for his help with the project. We are much indebted to Dr. D. Fesko for the many fruitful discussions and for supplying the samples.

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Discussion with Reviewers

Reviewer I: The authors use the references in an erratic way. E.g., "...the aluminized surface ... was defective⁵" implies that the reference determined that the authors's specimen was defective.

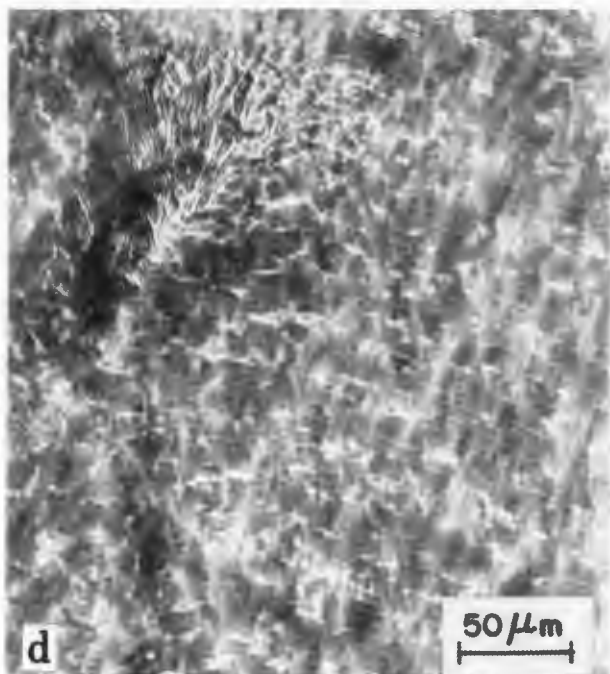
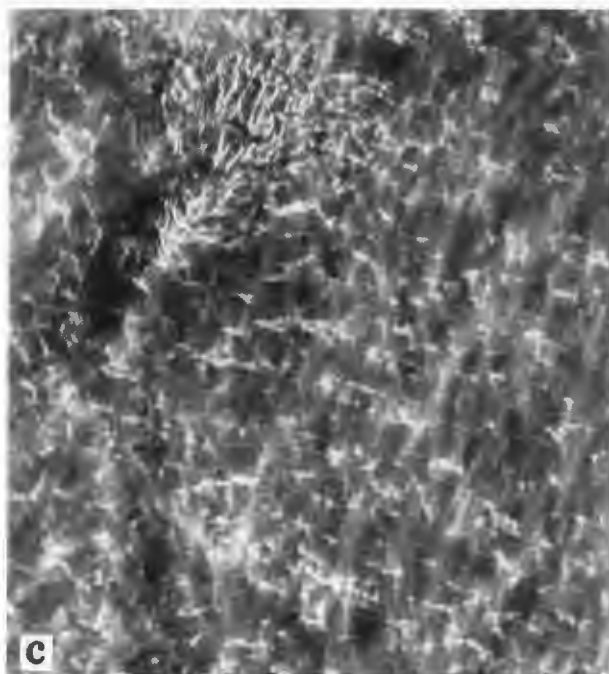
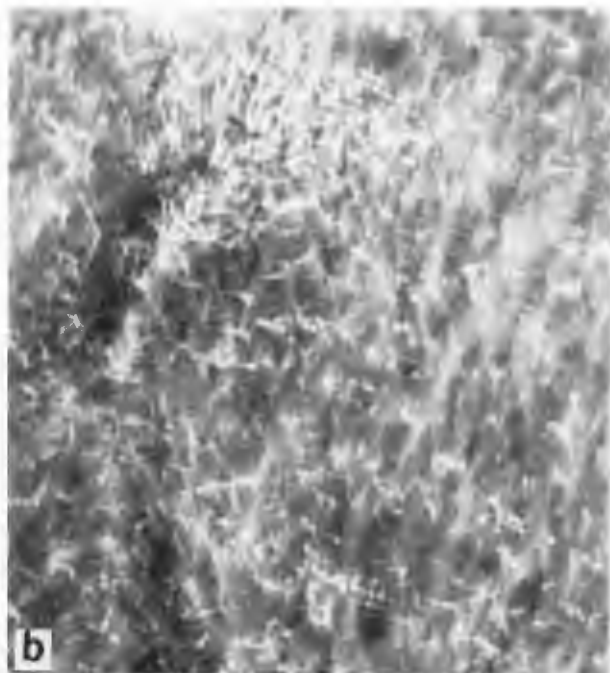
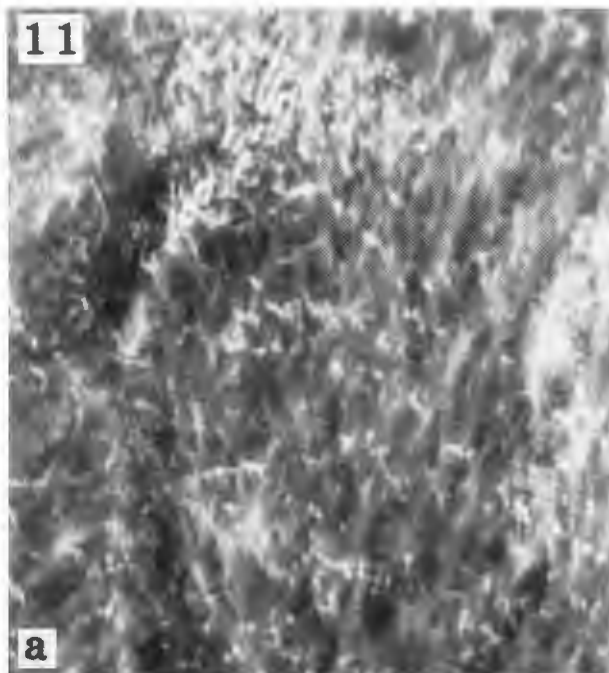
Authors: The use of reference 5 is to support our observations about the defects. We simply refer to similar observations by other researchers.

Reviewer I: Contribution of this paper: I believe that the authors proved that the base coat undergoes changes at temperatures that are used for curing the top coat. These changes apparently are due to a process of annealing the base coat that had been applied by smearing; thus, rheology-related stresses were probably annealed out, producing the corrugation. Elimination of the base coat gave no corrugation. The fact that the wrinkles are perpendicular to the direction of smearing of the base coat is convincing in proving the rheology connection.

On the other hand, I am not really convinced that the annealing of the polycarbonate base would produce corrugations AND interference colors; this is not proved or examined in this paper. Figs 11 e-h look interesting but they do not prove to me that the aluminum surface on such a substrate would give interference colors. The latter are the authors' main phenomenological problem. I do not see in Fig 11 h a corrugation such as in Fig. 1-3 or in Fig. 10.

It is also possible that the solvent contained in the base coat for the purpose of its application interacted with the polycarbonate base, sensitizing it toward a subsequent annealing during the curing operations.

Authors: The injection molded polycarbonate base has molded-in residual stresses and with annealing the surface will shrink and thus "wrinkle" the deposited aluminum film. The micrographs are incontrovertible evidence that interference colors result from stress relieving this surface. The density of the lines spacing is similar to a fine grating and it is no surprise that interference colors are produced. In addition, the last paragraph in the residual stress section explains our observations. These residual stress related experiments were conducted on samples without a base coat. Therefore, solvent interaction with the polycarbonate base is eliminated.



Reviewer I: In the Introduction, the second paragraph: "... indicating that stresses and not the base coat caused the defects." This is confusing, interfering with the assessment of the role of the base coat in other parts of the paper. The statement only holds for a case when NO base coat is present. Then, how could it affect anything if it is not present?

Authors: This paragraph has been taken out-of-context. The underlying cause/s for the

surface anomalies, at temperatures above the lacquer curing temperature, produced fine surface defects when no base-coat was present indicating that stresses and not the base coat caused the defects.

Reviewer I: Temperature effect section: "Some lines, however, lack continuity thickness³". Improbable and unwarranted; a simple random process of relaxation MUST occasionally produce such line ends.

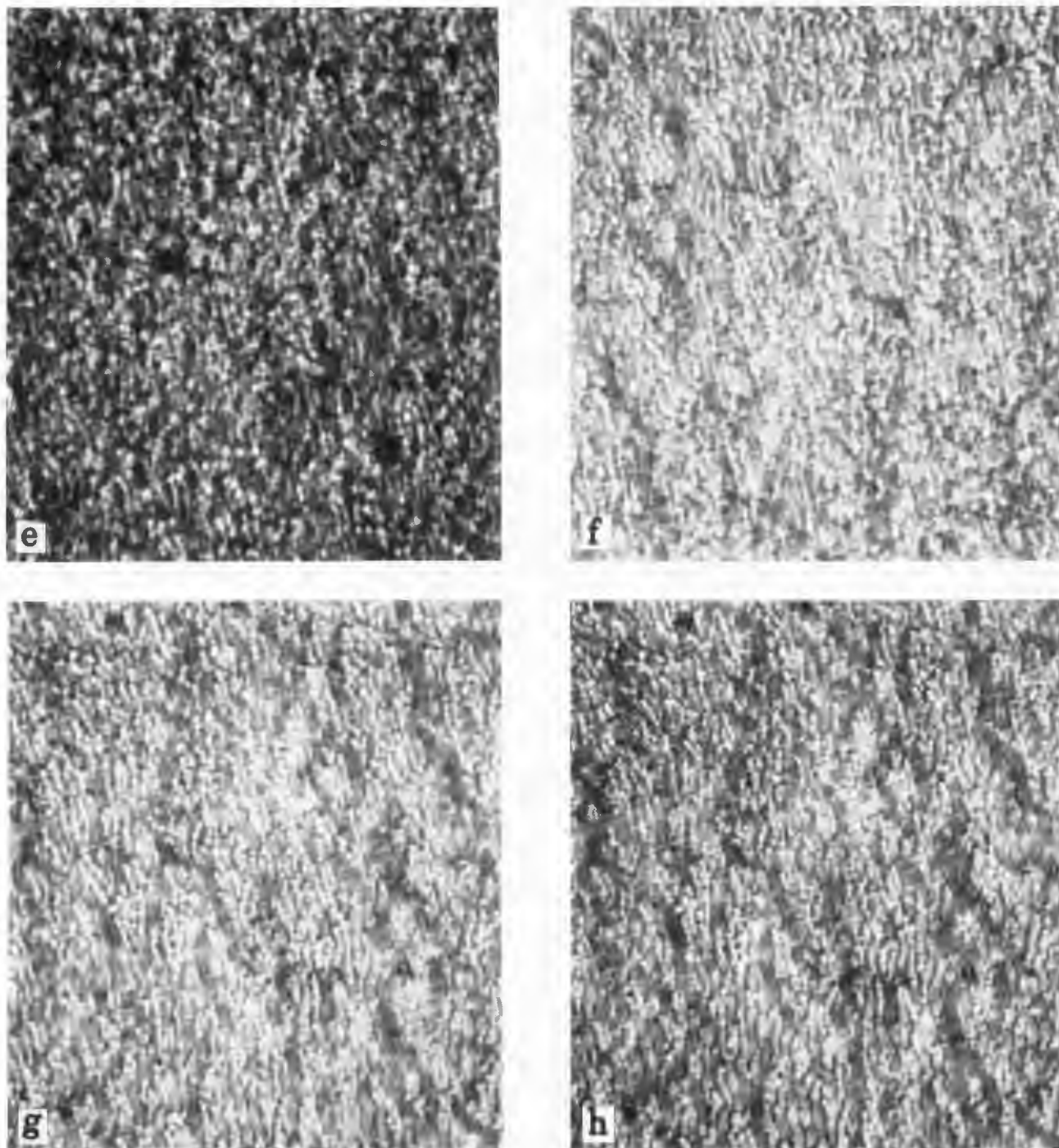


Fig. 11. Images resulting from the influence of residual stress and recorded during hostage experiments. Reduced (Figs. 11 a - d) and full wall thickness (Figs. 11 e - h) samples were used. All samples were uncured and without base coat. (a) 1 hour at 122 C; (b) 1.5 hours at 122 C; (c) 3 hours at 122 C; (d) 4 hours at 122 C; (e) 15 minutes at 122 C; (f) 30 minutes at 122 C; (g) 45 minutes at 122 C; and (h) 1 hour at 122 C. All figures are at same magnifications, note bar in Fig. 11 (d).

Authors: Nothing in our experiments indicated that only a "simple random process of relaxation must occasionally produce" - as the reviewer suggests - the observed line terminations. Indeed

not, because the process is very complex. Oversimplification will not suffice because the effect of the various factors such as the individual or combined influence of the base coat or

aluminum film thickness, the curing and the cooling temperatures and other, must be considered.

Reviewer I: Base Coat Curing Section: The effect of cooling rate. "Rate" is erroneous in this case. It is the temperature, not the rate that is studied here.

Authors: Room does not permit an in-depth discussion of the heating rate influence on the microstructure. If temperature is the only important factor, as the reviewer suggests, we would have missed observing the microstructures which developed in stages during the heating and cooling process.

Reviewer I: I am uncomfortable with the authors' use of "base coat curing". I assume that in the manufacturing process the base coat is not intentionally "cured." That is done to the top coat. I assume the base coat undergoes annealing.

Authors: The reviewer's assumption is incorrect regarding the base coat curing because the base coat is intentionally exposed to a cure temperature.