

CURE OF COATING COMPOUND POWDER
BY THERMAL IRRADIATION

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Analyzed are the structurization characteristics of a polymer coating compound during cure by various modes of heating.

In many branches of industry polymer coating compounds are now used whose cure quality does in many ways affect the operational reliability of costly components and devices. The process of curing these coatings by convective—conductive heating cannot be automated, because it is too slow.

Desiccation and cure of varnish—paint coatings by thermal irradiation has been known to be most effective, inasmuch as high-quality production can be achieved here while the process of curing becomes faster [1]. Infrared irradiation of polymer compounds for hermetic coatings is a process which can be highly automated in many technologies [2]. A study of the effect which thermal irradiation has on the structurization of polymer binders will, in turn, reveal the optimum conditions for the cure process.

At present compound powders are more widely used for coatings, and yet their structurization during radiative heating has hardly been explored at all [3].

The object of our study was a typical representative of these compounds, namely a filled polymer system comprising a mixture of grades ED-5 and ED-L epoxide resins with mineral fillers and a pigment. Specimens of this compound were cured in pans of metal foil as the substrate.

Various conditions of thermal irradiation or composite heating could be established in the apparatus, the latter having been designed to accommodate various kinds of infrared sources with the possibility of regulating the density of incident radiation flux (q_{23}) as well as the temperature (t_1) and the velocity (v_1) of the blowing air stream.

Transformations within the compound material during heating were indicated by an increase in the gel fraction, by a change in the amount of equilibrium swelling, and the shape of the thermomechanical curves.

In order to select the radiation sources properly, we evaluated certain optical characteristics of the given polymer system by the method shown in [4]. It has been established that maximum reflection of infrared energy occurs within the 3-5 μm range of the wavelength spectrum. The spectral reflectivity of this compound is minimum within the emission band of high-temperature quartz sources.

The penetrability of this filled polymer system is minimum to 1-2 μm wavelengths, with infrared radiation penetrating to a depth $\delta \approx 1000 \mu\text{m}$. It has been found, moreover, that infrared radiation beyond the 2.5 μm wavelength is strongly absorbed already in the surface layer of this compound. Thus, quartz sources seemed preferable for curing the system. Tests have also shown that the process is accelerated here, which, in our opinion, can be attributed to the higher vibratory energy of reactive epoxide groups (Fig. 1). As is well known, interaction between infrared radiation and a polymer material involves not only purely thermal effects but also implies a certain selective generation of radiant energy at the active atom groups of the cured material [5].

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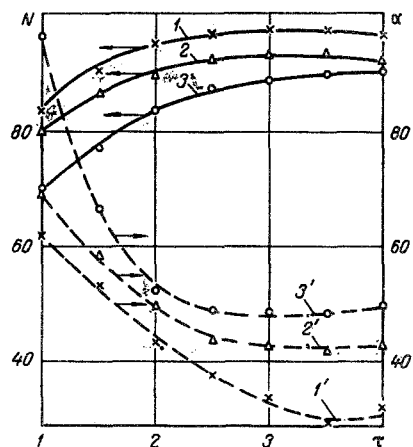


Fig. 1

Fig. 1. Solid residue content N (%) and equilibrium swelling α (%) in unextracted specimens, as functions of the curing time τ (h): convective heating (curves 3 and 3'), heating with quartz sources (curves 1 and 1'), and heating with tubular electric heaters (curves 2 and 2').

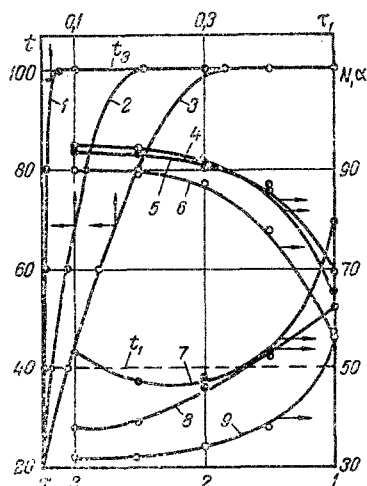


Fig. 2

Fig. 2. Solid residue content N (%) (curves 4, 5, 6) and equilibrium swelling α (%) (curves 7, 8, 9) as functions of the cure time τ (h), with heating times $\tau_1 = 3, 10,$ and 20 min (temperature-time curves 1, 2, and 3 respectively); air temperature t_1 ($^{\circ}\text{C}$).

It is evident, according to Fig. 1, that the structurization of this system (based on data pertaining to the buildup of solid residue and to equilibrium swelling) is accelerated by 20–30% (first stage of the process) and by 10–15% (period of quasisteady heating) when short-wave infrared sources (quartz sources) with maximum emission within the narrow 0.9–1.4 μm wavelength range are used rather than other heating methods, with identical averaged temperature-time characteristics of the thermal irradiation process for curing the compound.

We note that the curing of systems like the one studied here proceeds in several stages. During the first stage the material melts and polymerizes, whereupon the polymer molecules build up. The polymer structure and the conformation of its chains, as well as the supermolecular structures formed during the first stage of heating, play evidently an important role in producing the finished product. In view of this, we have considered the effect of two modal parameters especially important in the subsequent cure process during the first stage of heating already. These are the compound heating rate m , up to a certain quasiconstant temperature \bar{t}_3 , and the conditioned ambient temperature levels t_1 . The thickness of the specimens in this study was $\delta_3 = 2$ and 5 mm respectively. The prescribed quasiconstant specimen temperature ($\bar{t} = 100^{\circ}\text{C}$) was reached within various periods of time, by varying the density of incident radiation flux q_{23} . The test data are shown, averaged, in Fig. 2.

According to Fig. 2, the rate at which a coating was heated had an appreciable effect on the final compound structure. As the system heating rate m was changed from 27 to $4^{\circ}\text{C}/\text{min}$, the structural defectiveness α decreased also appreciably (from 54 to 32.7% respectively). We have the following explanation for this. In a compound powder during thermal irradiation there can initially occur two processes, as has been mentioned earlier, namely melting of the binder and its polymerization. In this case, when the system is heated rather slowly ($\tau_1 = 20$ min) to the prescribed temperature $\bar{t}_3 = 100^{\circ}\text{C}$, the curing process is also rather slow. This period is characterized by conformational transformations of the binder molecules, as a result of which the polymer molecules move into favorable relative positions so as to produce the densest possible lattice. When the system is heated fast ($\tau_1 = 3$ min), then many more polymerization centers emerge here and the system seems to set at many locations. Its mobility is thus reduced and there is not enough time for the completion of the relaxation processes involved in the regrouping of molecules as well as in the transition of the system from one state to another, which then leads to a defective lattice.

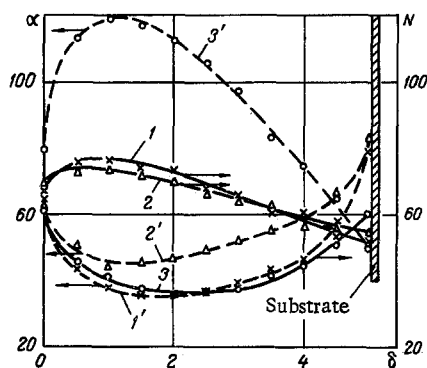


Fig. 3

Fig. 3. Solid residue content N (%) and equilibrium swelling α (%) across the specimen thickness δ (mm), during convective heating (curves 3 and 3'), during heating with quartz sources (curves 1 and 1'), and during heating with tubular electric heaters (curves 2 and 2').

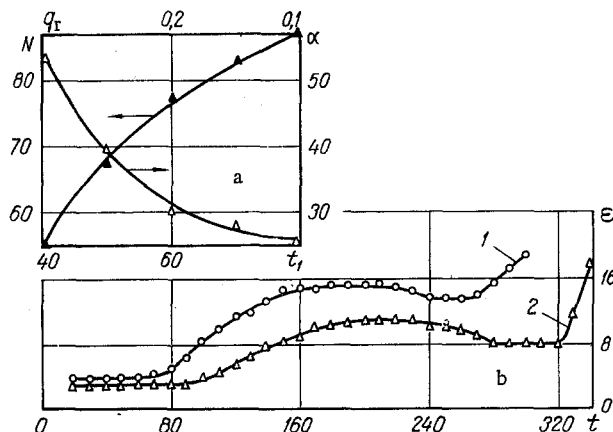


Fig. 4

Fig. 4. (a) Solid residue content N (%) and equilibrium swelling α (%) as functions of the air temperature t_1 ($^{\circ}\text{C}$) and of the radiation flux density q_R (W/cm^2); (b) thermomechanical curves for specimens cured by convective heating (curve 1) and by thermal irradiation (curve 2): strain ϵ (%), specimen temperature t ($^{\circ}\text{C}$).

It was also of interest to explore the effect which the characteristics of the various heating modes, at the same rate, have on the polymer structurization. For this purpose, the specimens (the 5 mm high cylinders with plane-parallel end faces) of cured compound were examined sectionwise. Layers of the compound were sliced off accurately within 0.0125 mm on a model TV-4 cutter machine. The cutting speed was such as to preclude the destruction of a specimen during the operation.

It is evident, according to Fig. 3, that the structurization of a coating proceeds differently as result of convective heating and by thermal irradiation. The fact is that the initial orientation of polymer molecules, especially in evidence in the boundary layers, appreciably affects the structurization kinetics and dynamics in coating compounds, depending on the specific mode of heating during the subsequent cure stages. We note that, accordingly, this affects also the thermophysical properties of the system and causes differences in the resulting temperature field of a coating, even though the averaged "heating curves" are the same [6, 7]. During convective heating, the highest temperatures $t_{3\text{max}}$ (and the maximum cure) in a coating are noted in the surface layers. During thermal irradiation, on the other hand, $t_{3\text{max}}$ occurs in some inner layer, depending on the optical permittivity characteristics of the system.

Considering that infrared radiation had been found to penetrate the compound specimens to a depth $\delta \approx 1$ mm, we examined the conditions of combined radiative-convective heating which would ensure the most favorable structurization of the system. The level of cure in a hermetic coating was highest, according to Fig. 4a, with the ambient hot air at the temperature $t_1 = 70^{\circ}\text{C}$ * and the density of radiation flux $q_{23} = 0.15 \text{ W}/\text{cm}^2$. This was entirely feasible, considering that, under the given conditions of combined radiative-convective heating, the temperature was highest in the inner layers of the coating. In this case, however, both conductive heat leakage to the outer layers and convective heat transfer to the boundary layer of the coating had played an important role in shaping the temperature field. Thus, according to our calculations, the convective component of the radiative-convective heat transfer contributed approximately 25% to the total heat balance in a closed adiabatically heated volume of the coating-substrate system at $\bar{t}_3 = 100^{\circ}\text{C}$. Such a conditioned radiative-convective heating of a powder-substrate system would ensure a sufficiently uniform temperature field and a sufficiently high cure rate.

In order to verify the preceding analysis, the same compound was cured comparatively by various heating modes within equal time periods at the same average specimen temperature. The specimens were then analyzed on the basis of thermomechanical curves plotted during uniaxial compression under a constant load of $33.4 \text{ kgf}/\text{cm}^2$. These data are shown in Fig. 4b. According to those curves, the glass transition

* The blow velocity within the range $v_1 = 0.25-0.50 \text{ m}/\text{sec}$ did not significantly affect the cure kinetics.

temperature and the breakdown temperature of the compound cured by radiative--convective heating (curve 2) were higher but the superelastic strain was much lower than in the case of convective curing (curve 1). During identical average temperature--time cycles, therefore, the structural transformations were more thorough in specimens subjected to radiative--convective heating.

Thus, our study has established the complex effects of radiative--convective heating on the physico-chemical transformations in a filled polymer system, and has revealed the advantages of thermal irradiation over convective heating for curing this particular compound.

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