*A Method for Casting Thin Epoxy Films**

INTRODUCTION

Many studies of epoxy resins require specimens in the form of thin films, on the order of tens of micrometers thick. For example, thin films are necessary for transmission IR studies and certain diffusion experiments where rapid equilibration is desirable. Our application required thin-film epoxy specimens with smooth surfaces and known surface area.

Fabrication of cured epoxy samples in the form of thin films is difficult. Cured epoxies are too frangible for microtomy, and casting between salt plates,[1] while useful for IR studies, is undesirable because the expensive salt plates must be dissolved to obtain the samples. We report here a method for fabricating films of cured epoxy resin on the order of 10 μm thick by casting between two flat, silicone rubber platens. The silicone rubber requires no release agent and imparts a smooth surface finish to the cast films, as reported by Fanter.[2] This note discusses the techniques both for fabricating the silicone rubber platens and for casting the epoxy films.

PREPARATION OF FLAT SILICONE RUBBER PLATENS

The substrate consisted of a piece of 6-mm-thick plate glass approximately 30-cm square, frosted on one side. Any material would suffice for a substrate provided that the surface is rough enough to promote adhesion of the silicone rubber and sufficiently rigid to support the resulting sheet. Approximately 200 g of the silicone rubber compound (General Electric RTV 664) was mixed and deaerated according to the instructions provided with the RTV, and then poured onto the frosted substrate surface. Four spacers, 3 mm thick, were placed on the substrate to establish the thickness of the RTV sheet, (see Fig. 1). A piece of smooth plate glass was then pressed against the RTV to form the smooth, flat surface. The silicone rubber was allowed to cure at room temperature for 24 h; then the plate glass was gently removed, leaving the 3 mm thick RTV layer adhered to the frosted glass substrate. Separation of the smooth plate glass from the cured RTV sheet was accomplished by using a wooden ruler as a long wedge and gently driving it between the glass plates of the sandwich. Two such glass substrate–silicone rubber platens were prepared and post-cured in air at 177°C (the highest temperature in the cure/post-cure cycle for the epoxy) for 12 h.

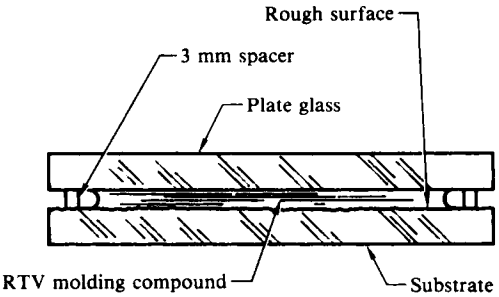

Fig. 1. Details of sandwich for fabricating silicone rubber platens.

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CASTING OF EPOXY THIN FILMS

Both the glass-silicone rubber platens and the epoxy resin (Ciba Geigy MY720 mixed with Eporal) were heated to 150°C, at which temperature the resin initially has a low viscosity. A spatula was used to place small, typically 1 mm diam, drops of the resin spaced approximately 5 cm apart on one of the silicone rubber platens. After the platen's surface was covered with drops of the resin, the second heated platen was placed on top to form a sandwich as shown in Figure 2. The top platen was rotated slightly and pressure was exerted to squeeze the two platens together and flatten the resin droplets. The whole assembly was then placed in the curing oven, and the epoxy cure/post-cure temperature cycle was completed.

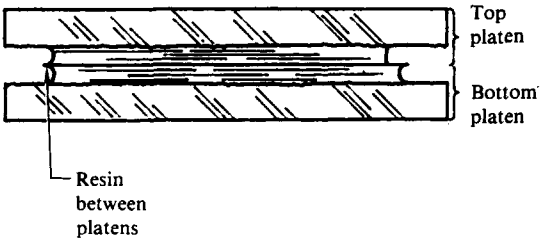

Fig. 2. Details of sandwich for casting thin epoxy films.

After curing, the two platens were separated using a wooden ruler as a lengthwise wedge as described above. Almost all the thin films of epoxy resin adhered to one of the platens. Removal of the individual pieces of thin epoxy film was accomplished with a tapered-end flat spatula. The tapered end of the spatula was pressed against the silicone rubber surface of the platen and gently forced under the thin film of epoxy. After the point of the spatula was under the epoxy film, a smooth sideways motion of the tapered end separated the film from the silicone. Development of the appropriate technique required some practice before samples could be removed intact.

SUMMARY

Thin disks, typically 1 cm diam, of cured epoxy films with thicknesses ranging from 10 to 40 μm were obtained by this casting method. Variation in thickness across a single piece of film was less than 10%. No effort was made to control the thickness of the pieces of cured resin. Depending on the size of the resin droplet and the care exercised in removing the thin epoxy film, samples of larger surface area could probably be obtained. This technique may not be suitable for surface studies because organosilicones may adhere to the sample surfaces.

References

1. W. J. Price, *Laboratory Methods in Infrared Spectroscopy,* 2nd ed., R. G. J. Miller and B. C. Stace, Eds., Heyden, New York, 1972, p. 113.
2. D. L. Fanter, *Rev. Sci. Instrum.*, 49(7), 1005 (1978).

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