Microstructure and Properties of Epoxy Foams Prepared by Microwave

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ABSTRACT: In this article, epoxy foams comprised of diglycidyl ether of bisphenol-A (DGEBA) based epoxy resin E_{31} and E_{51}, polyamide resin, and water were prepared by microwave irradiation method. The structure and properties of epoxy foams were analyzed by FTIR, TGA, SEM, and DMA methods. The density and compressive performance of epoxy foams was also determined. The results indicated that the epoxy foams had excellent compressive performance and the preparation of epoxy foam by microwave irradiation was high efficiency and convenient. The composition has great effect on density, foam structure, dynamical mechanic performance, and thermal degradation behavior of epoxy foams. The epoxy foam with density from 0.08 g cm^{-3} to 1.05 g cm^{-3} can be obtained by varying ratio of E_{51} and E_{31} to control the viscosity of mixtures. J Appl Polym Sci 112: 3543–3547, 2009

Key words: microwave irradiation; epoxy foam; compressive performance; microstructure

INTRODUCTION

Relative to traditional heat processing polymer materials method, microwave processing is a new technology developed rapidly in recent 20 years, which provides new technology to improve the physical properties of materials; alternatives for processing materials that are hard to process; a reduction in environmental impact of material processing; economic advantages through energy savings, space, and time; and an opportunity to produce new materials and microstructures that cannot be achieved by other method.[1] Compared with conventional method, microwave processing has many advantages such as penetrating radiation, controllable electric field distribution, rapid heating, selective heating of materials, and self-limiting reactions.

Polymer foams such as silicone foams and food foams were prepared by microwave technology with microwave active materials and water as foaming agents.[2–5]

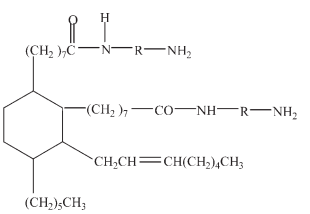
Epoxy bulk materials has excellent mechanical and processing properties, the preparing method of epoxy foams was investigated by many engineering scientist. One of the most significant applications for syntactic foams is in the area of naval and undersea marine equipments, where it has been used as structural element for decks and submarines buoys. Syntactic foams were prepared by using glass, ceramic, or polymer micro-balloons as fillers in epoxy matrix,[6–9] but this method cannot obtain foams with lower density and micro-balloons cannot be dispersed homogeneously because of different density of epoxy resin matrix and fillers and the range of volume fraction of fillers could be adjusted between 0% and 45%.

In this article, for the first time, we reported a new method of pure epoxy foams prepared by microwave irradiation. Epoxy foams with low molecular weight diglycidyl ether of bisphenol-A (DGEBA) epoxy resin (E_{31} and E_{51}), polyamide resin as curing agent, and water as foaming agent were prepared by microwave irradiation method, and the performance and properties of foams were analyzed.

EXPERIMENTAL

Materials

In this study, bisphenol A epoxy resin with epoxy value equaled to 0.31 and 0.51 according to E_{31} and E_{51}, respectively, was supplied by Yueyang chemical plant, China; low molecular polyamide resin with trade name PA651 (amine value = 300 mg KOH g^{-1}) was supplied by Tianjing Yanan chemical plant, China; distilled water. The chemical structure of polyamide resin is shown as follows:



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Preparation of epoxy foams

E_{31} and E_{51} epoxy resins with different ratio, polyamide resin, and one or two drops of water with total weight about 30 g were added in a paper cup and mixed homogeneously with electric stirrer. A few minutes later, the mixtures were placed in microwave oven with a fixed frequency of 2.45 GHz and processed for 30 s to 1 min. The mixtures was cured and foamed simultaneously after 1 or 2 min.

Analytical techniques

The IR analysis was performed by Nicolet-800 infrared spectrometric analyzer and the microstructure of

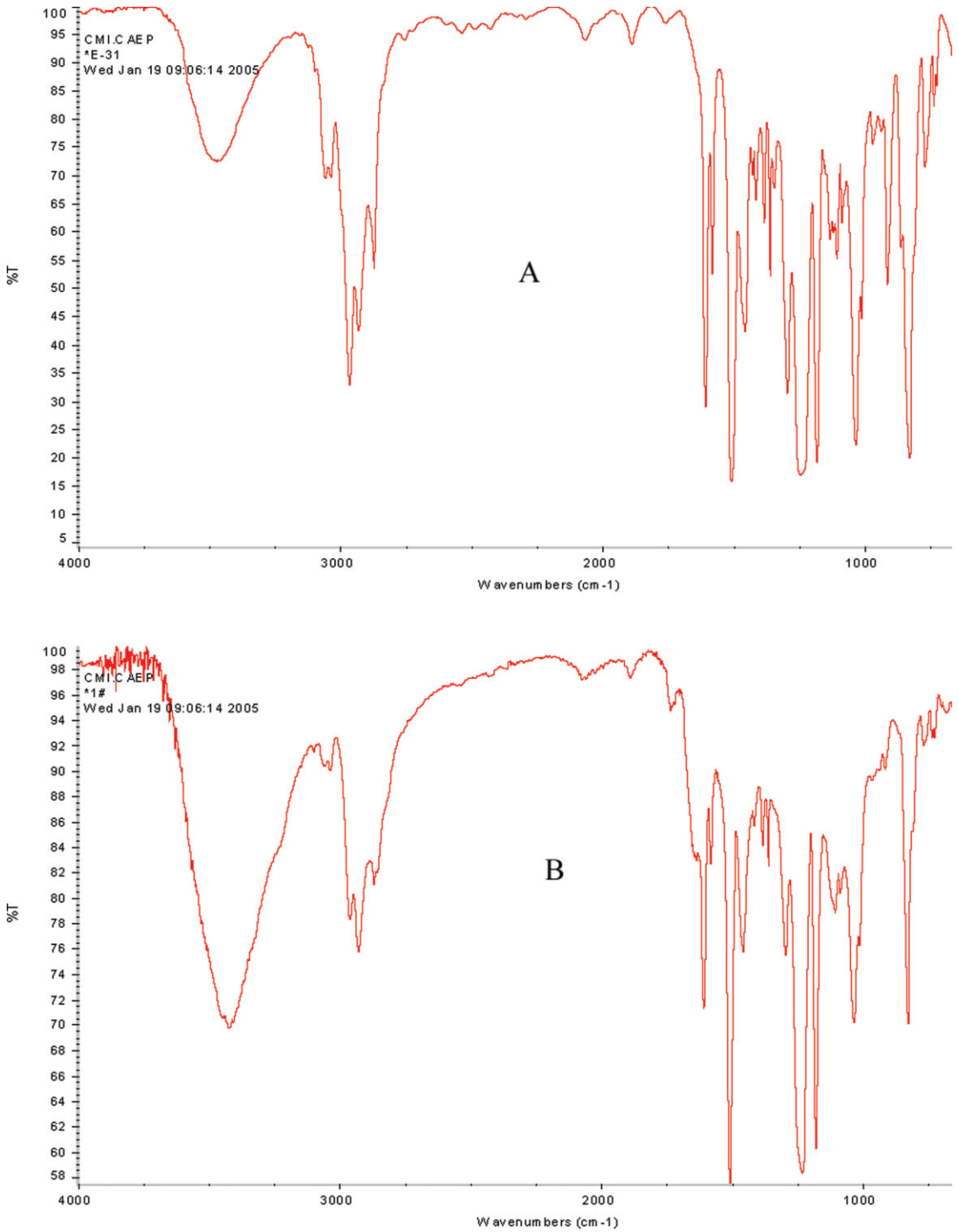

Figure 1 The IR spectra of epoxy resin (E_{31}) and epoxy foam (E_{51}/E_{31} = 50/50). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

TABLE I
Compression Performance of Epoxy Foams

Sample	Density (g cm^{-3})	Yield strength (MPa)	σ 0.01 (MPa)	Modulus (GPa)
1	0.37	6.90	2.17	0.23
2	0.43	12.26	5.99	0.63
3	0.57	18.81	8.86	0.94
4	0.62	23.22	10.90	1.14
5	0.99	73.28	29.20	2.91
6	1.05	83.32	40.10	3.26
7	1.07	84.84	32.90	3.32
8	1.10	96.88	35.20	3.48
9	1.15	105.50	39.10	3.94

foams was probed by SEM (AMRAY, China). The dynamical mechanical properties of epoxy foams were tested by Perking–Elmer DMA 7 Series and compression performance was determined by Instron 558 material testing machine with standard method; TGA curves were obtained by 2050 TGA instrument (TA corp.).

RESULTS AND DISCUSSION

IR spectra of epoxy foams

The spectra of epoxy foam and DGEBA epoxy resin (E_{31}) were showed in Figure 1. The peak at 830 cm^{-1}, 1250 cm^{-1}, and 1040 cm^{-1} were phenyl ether, substituted benzene and aliphatic ether in DGEBA epoxy resin. The peak at 915 cm^{-1} in spectra A is unreacted epoxy group in epoxy resin, comparing to A, this peak is disappeared in spectra B (epoxy foam), indicated that epoxy group is completely reacted with amine group of polyamide resin.

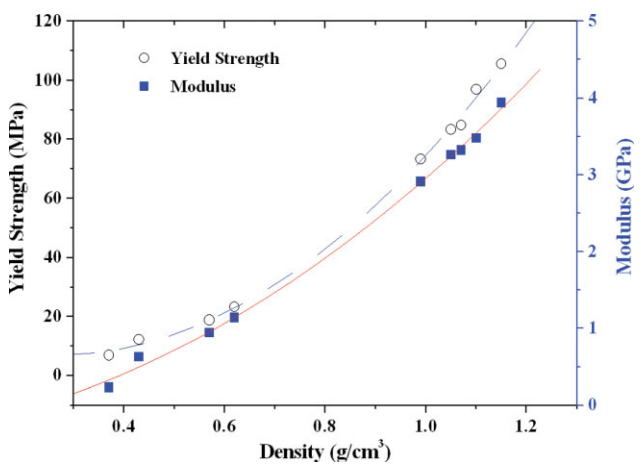

Figure 2 Yield strength and modulus of epoxy foams varied with density. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

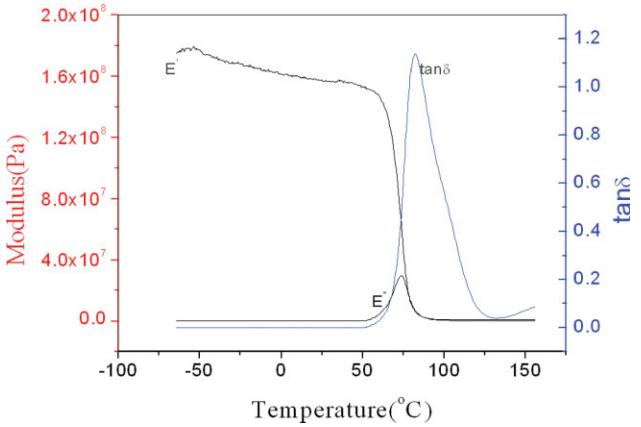

Figure 3 The DMA curves of epoxy foams (density = 0.37 g cm^{-3}). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Mechanical characterization of epoxy foams

Generally, the mechanical performance of porous materials varied with its density. The compression performance of epoxy foams with different density was listed in Table I. It must be noted that the densities of epoxy foams approach to 1.0 g cm^{-3} (such as samples from 6 to 9 in Table I) are not really foams, although the density of epoxy bulk material approaches to 1.2 g cm^{-3}. The result showed that the modulus, yield strength, and σ 0.01 were increased with the increase of density (Fig. 2). The mechanical performance of epoxy foam with density at 0.5 g cm^{-3} is better than that of polyurethane foam with same density.[10]

Dynamic mechanical properties of epoxy foams

The dynamic mechanical properties of epoxy foams were showed in Figures 3 and 4. The results showed

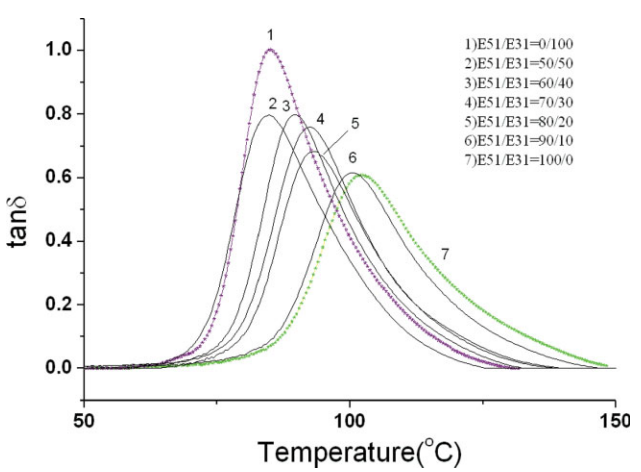

Figure 4 The Tanδ-T curves of epoxy foams. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

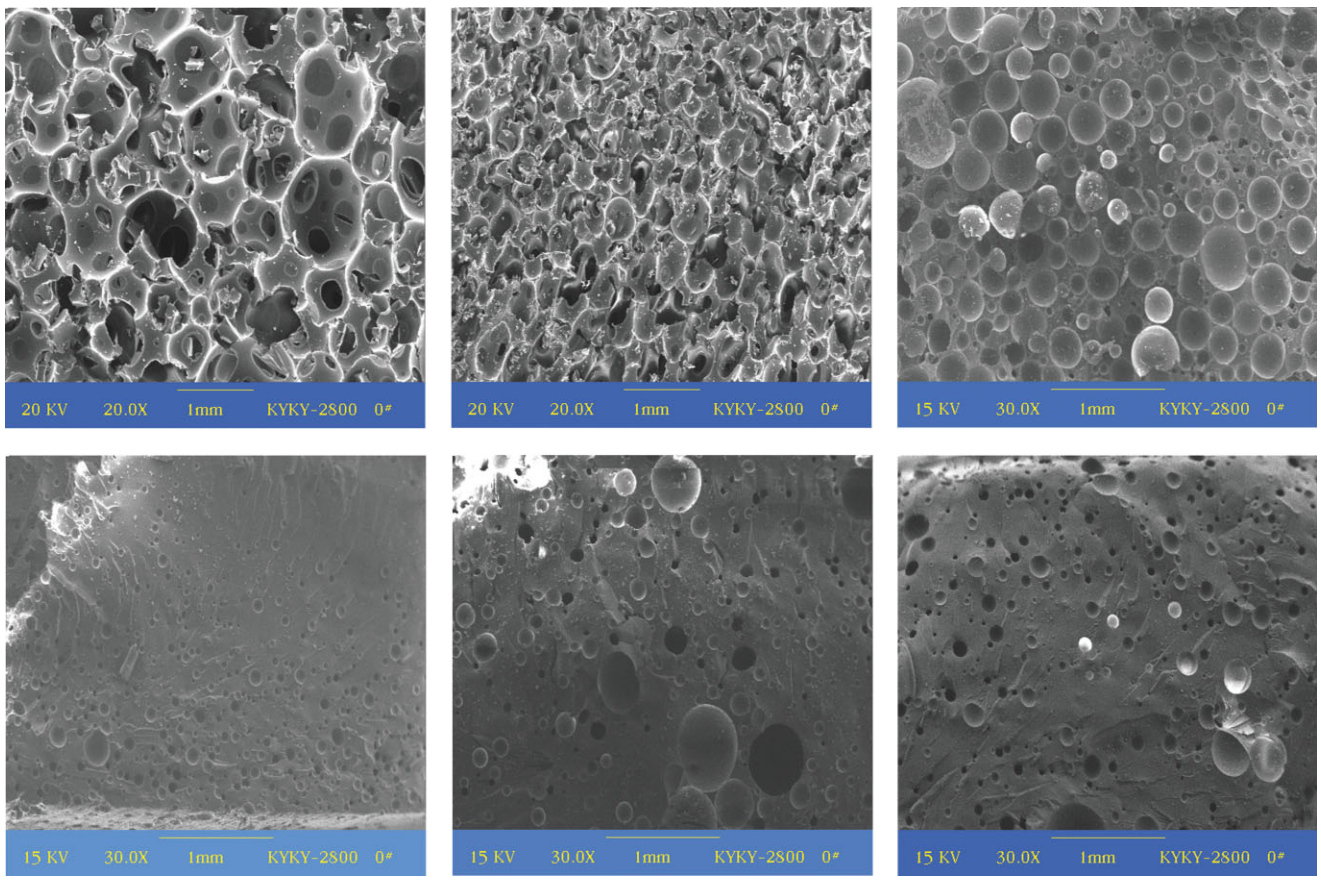

Figure 5 SEM photographs of epoxy foams with different density. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

that the glass transition temperature of epoxy foams is lower than 100°C, much lower than that of epoxy bulk material, whose glass transition temperature is about 120°C. Like polymer bulk materials, viscoelastic polymer foams also have damping performance. The results indicated that glass transition temperature of epoxy foams was shifted to higher temperature along with the increase of E_{51} content; at the same time, the Tanδ maximum value became smaller because of E_{31} epoxy resin has higher molecular weight than E_{51} epoxy resin. The broaden glass transition temperature also implied that damping properties of epoxy foams would be excellent, as well as other polymer foams.

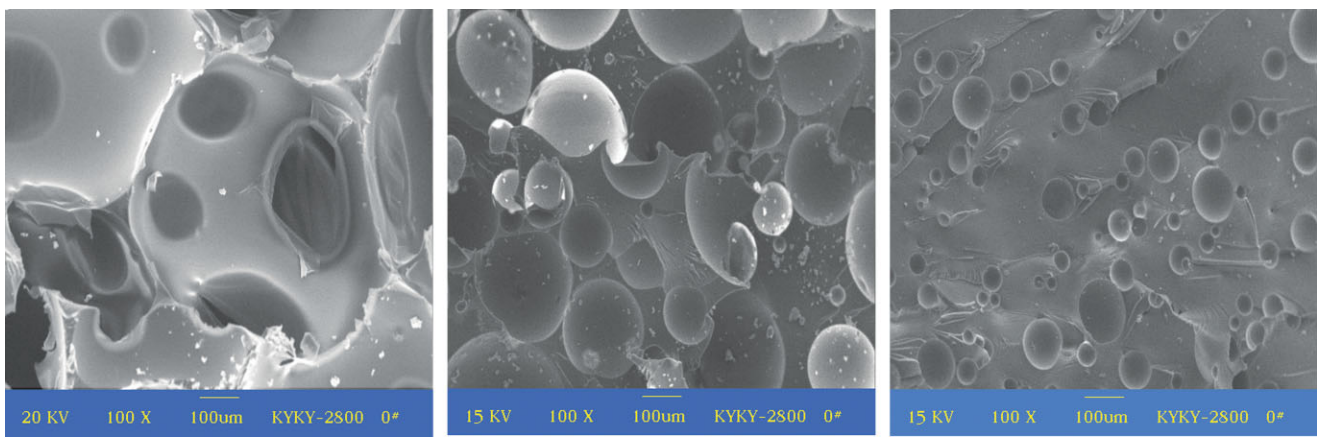

Figure 6 The cell structure of epoxy foams. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

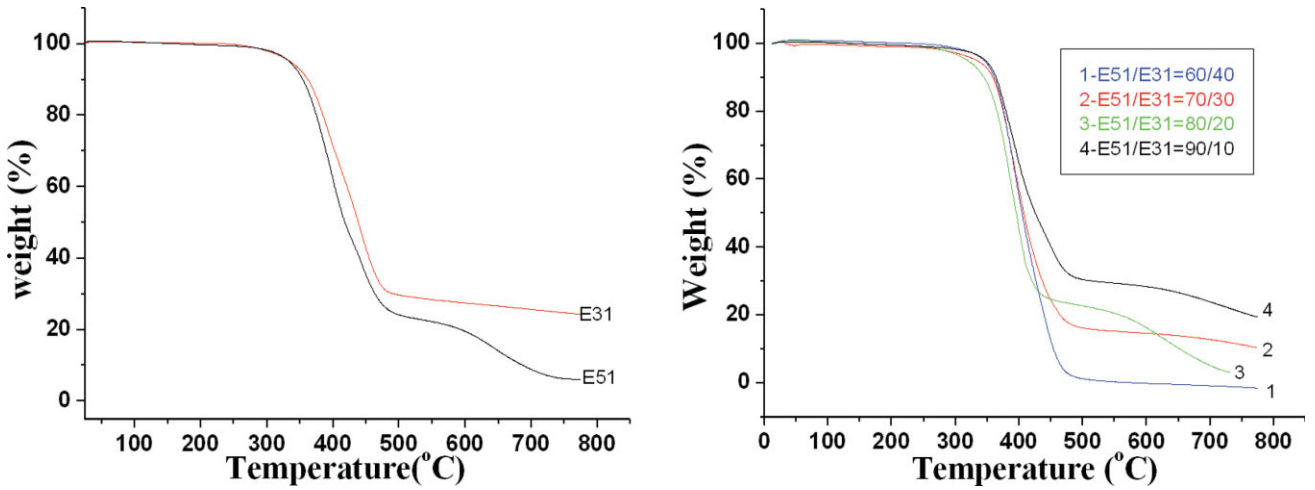

Figure 7 The TGA curves of epoxy foams. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

The microstructure of epoxy foams

The mechanical performance, dynamic mechanical performance, damping property, and thermal conductivity of foams are seriously affected by cell structure. The microstructures of epoxy foams with different density were showed in Figure 5.

With increasing of the content of E_{31}, the viscosity of mixtures increased, the foaming of mixtures was inhibited. The epoxy foams have open-cell structures with density lower than 0.52 g cm^{-3}, but when the density is higher than that value, the foams have close-cell structures (Fig. 6).

Thermostability of epoxy foams

The epoxy foams have good thermostability, the start thermolysis temperature is above 250°C as shown in Figure 7. Although epoxy resin E_{31} and E_{51} have same chemical structures, but the foams with different ratio of E_{31} and E_{51} have different thermolysis behavior because the molecular chain of E_{31} was longer than that of E_{51}, so the higher the content of E_{31} the better thermostability of the foams.

CONCLUSION

Using water as foaming agent, pure epoxy foams comprised of DGEBA based epoxy resin and low molecular weight polyamide resin as curing agent could be prepared conveniently and rapidly by household microwave oven.

The curing reaction of epoxy foams was completely in the condition of experiment and thermostability, mechanical performance, and dynamic mechanical properties of epoxy foams was quite well. The epoxy foams with density from 0.08 g cm^{-3} to 1.05 g cm^{-3} could be obtained easily by adjusting the viscosity of the reaction mixtures with the content of epoxy resin E_{31}. The microstructures of epoxy foams changed from open-cell structure to close-cell structure with the increase of density.

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