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Temperature and radiation effects on the Raman bands of epoxy resin

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Abstract. The C–H stretching, in-plane bending and out-of-plane bending modes of epoxy resin are investigated using Raman spectroscopy. Changes in Raman frequencies show a weakening of the hydrogen bonds on heating, but no effect on irradiation up to a total dose of 16 Mrad.

Keywords. Raman spectra of epoxy resins; temperature and irradiation effects.

1. Introduction

The effect of temperature on phenoxy resin (Coleman and Moskala 1983) and the effects of solvent and temperature on epoxy resin (Janarthanan and Thyagarajan 1988) has been studied by FTIR spectroscopy. In both cases, the band at about 3400 cm^{-1}, attributed to a wide distribution of hydrogen-bonded hydroxyl-stretching frequencies, shifts to higher frequencies, indicating a weakening of the hydrogen bonds. The aim of the present work is to study the temperature and radiation effects on some of the Raman bands of epoxy resin.

2. Experimental

The epoxy resin used in this study was obtained from Ciba-Geigy. The Raman bands were recorded using a 4-watt Argon ion laser and a Jobin–Yvon Ramanor spectrometer. The slit width used was 220 μ and the spectra were recorded with a scan speed of 20 cm^{-1}/min. Before recording the spectra, the sample was exposed to laser beams for three hours to quench the fluorescence. A temperature cell was used for high temperature studies with an accuracy of $\pm$ 1°C. For irradiation studies, the sample was irradiated in a γ-ray chamber by a ^{60}Co source with a dose rate of 0·39 Mrad/h. The sample was irradiated with a total dose of 16 Mrad.

3. Results and discussion

The prominent bands at 3080, 1262 and 831 cm^{-1} were studied. The O–H stretching frequency expected at about 3400 cm^{-1} as in IR is probably very weak and could

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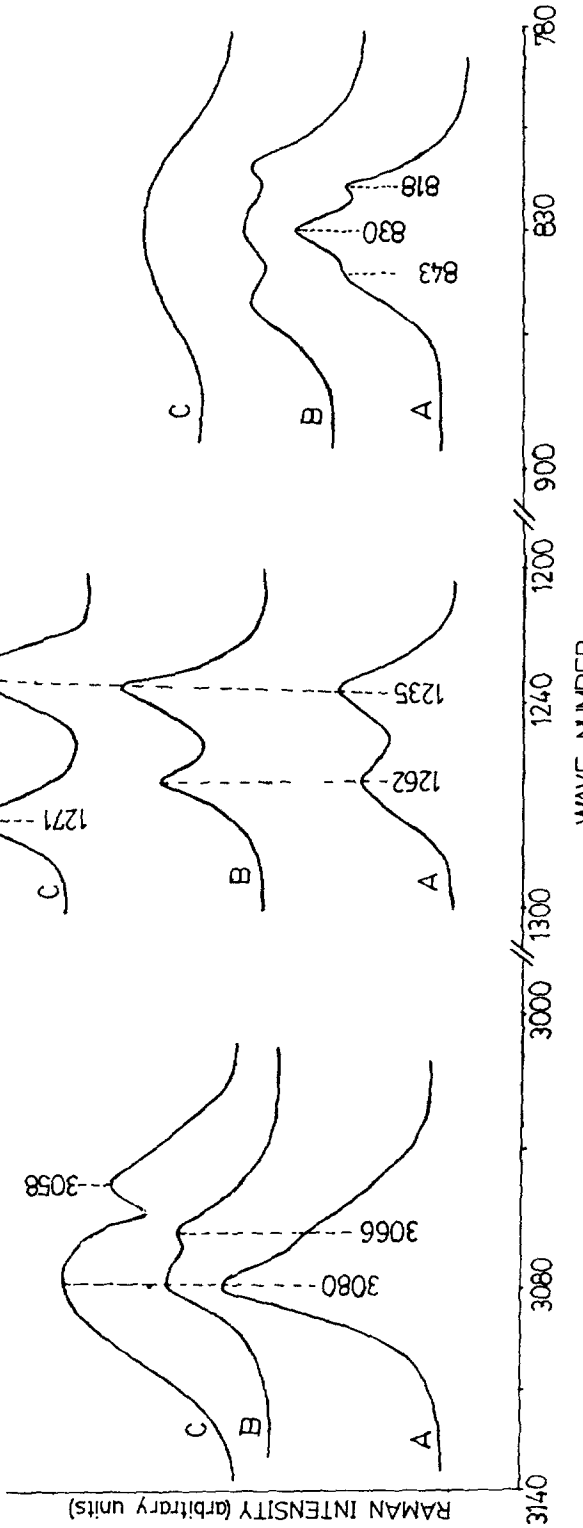

Figure 1. Raman spectra of epoxy resin in the regions (**a**) 3000–3140, (**b**) 1200–1300, and (**c**) 780–900 cm^{-1}. Curves A, B and C represent spectra recorded at room temperature, 75° and 125°C, respectively.

not be recorded. Figure 1a shows the Raman spectrum of epoxy in the region 3000–3140 cm^{-1} recorded at room temperature, 75°C and 125°C (curves A, B and C respectively). It has been observed that the band at 3080 cm^{-1} assigned (Martzel and Koenig 1986) to the stretching of the phenyl–hydrogen bond undergoes reduction in intensity as the temperature increases. Further a shoulder at 3066 cm^{-1} develops distinctly which shifts to lower frequency (figure 1c) as the temperature increases indicating the weakening of the phenyl–hydrogen bond.

In figure 1c the Raman spectra recorded in the region 780–900 cm^{-1} at room temperature, 75 and 125°C (curves A, B and C respectively) are shown. Three bands with peaks at 818, 830 and 843 cm^{-1} attributed to the phenyl–hydrogen out-of-plane bending are distinctly observed at room temperature. As the temperature increases to 75°C, the bands become broader and the peak intensity is reduced. At 125°C, the bands merge and give a very broad profile with much reduced peak intensity. This indicates the sensitivity of the out-of-plane vibrations to temperature changes.

In figure 1b, the bands in the region 1200–1300 cm^{-1} are shown. At room temperature, there are two bands at 1235 and at 1262 cm^{-1} (curve A). The band at 1235 cm^{-1} is assigned to the phenyl–hydrogen in-plane bending while the one at 1262 cm^{-1} is attributed to the epoxy ring (Martzel and Koenig 1986). It may be noted that the band at 1262 cm^{-1} is sensitive to temperature changes as can be seen from the frequency shift and intensity increase (curve c) whereas that at 1235 cm^{-1} shows only an increase in intensity but no shift in frequency. Since the changes in frequency and intensity in all the bands are indicative of the weakening of the bands, the origin of the changes can be traced to decomposition rather than structural changes.

The epoxy resin was irradiated by γ-radiation to a total dose of 16 Mrad in air. The Raman profiles of the irradiated sample showed no changes implying that the high energy radiation does not have any effect on the sample for a dose rate of 16 Mrads.

References

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