

特異な分解反応を利用するアクリル系硬化樹脂の
硬化反応および架橋ネットワーク構造の解析 (I 報)

反応熱分解ガスクロマトグラフィー

Characterization of Curing Reaction and Cross-linking Network Structures
in Cured Acrylic Resins Using Specific Decomposition Reactions (Part I)

Thermally-assisted Hydrolysis and Methylation Gas Chromatography

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Abstract

Chemical structures in acrylate-type ultra violet (UV)/electron beam (EB)-cured resins were characterized using specific sample decomposition. At first the UV cured resin samples were analyzed by thermally-assisted hydrolysis and methylation (THM) gas chromatography using tetramethylammonium hydroxide. Not only the products originated from the constituent monomers and prepolymers, but also methyl acrylate (MA) and its oligomers (up to hexamers) reflecting the cross-linking sequences, which were formed through THM reaction selectively occurred at ester linkages in the cured resins, were observed in the pyrograms. These products were interpreted in terms of the chemical composition, molecular weight of the constituent monomers (prepolymers), their double bond conversion in curing, and the sequence distributions of network junctions. Moreover, the UV cured resins were also characterized by matrix-assisted laser desorption/ionization mass spectrometry (MALDI-MS) combined with supercritical methanolysis of the samples. The methanolysis products were fractionated by size exclusion chromatography followed by MALDI-MS measurements of the individual fractions. The observed distributions of the decomposition products suggested that the network junctions in the UV-cured resin were composed of up to around 2,000 acrylate units. Moreover, the network structures even in more complex copolymer type UV/EB cured resin samples were also successfully characterized by this approach using high-resolution MALDI-MS.

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