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USAAVLABS TECHNICAL NOTE 5

A METHOD OF OBTAINING A TRUE INFRARED SPECTRUM OF THE EPOXY FROM A CURED FIBERGLASS-EPOXY COMPOSITE

By

James L. Perkinson, Jr.

August 1970

U. S. ARMY AVIATION MATERIEL LABORATORIES
FORT EUSTIS, VIRGINIA

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ABSTRACT

A method of separating epoxy from a cured fiberglass-epoxy composite has been developed and evaluated. This separation is necessary if a true infrared spectrum of the epoxy is to be obtained for the purpose of characterization. The method consisted of separating two materials of different specific gravities with a liquid of intermediate specific gravity - the concept of differential flotation.

Infrared spectra of three differently prepared specimens of the same lot of prepreg composite were compared. The resulting infrared spectra prove the feasibility of this approach to cured fiberglass-epoxy characterization.

TABLE OF CONTENTS

INTRODUCTION

Cured fiberglass-epoxy composites have been difficult to identify or characterize heretofore by infrared spectrophotometry because the ground fiberglass interfered with the analysis. Therefore, it was proposed that a method of separating the epoxy from a cured fiberglass-epoxy composite be developed in order to accurately analyze the chemical makeup of the epoxy.

The separation process evaluated in this note consists essentially of grinding a sample of the parent material and separating the constituents through differential flotation, thus permitting characterization.

SPECIMEN PREPARATION

To prove the feasibility of this separation method, three specimens were prepared from a single piece of uncured Scotchply 1002 prepreg roving. The first specimen was prepared by separating the constituents through a process of leaching the uncured epoxy-catalyst matrix from the fiberglass composite with methyl ethyl ketone ($CH_3COC_2H_5$) (MEK). The second specimen was prepared in a similar manner to the first except that the fiberglass was put back into the uncured leach mixture. The third specimen was prepared under normal conditions of layup - the roving was applied to the mold at the correct fiber angle, and heat and pressure were applied. The three specimens were cured at 280°F for 16 hours.

The purpose of specimen 1 was to obtain a spectrum of the pure epoxy after curing and, since it had no glass, to compare it with specimens 2 and 3 to determine if the heat of friction from grinding the glass had any effect on the characteristics of the infrared spectrum.

The purpose of specimen 2 was to act as a check between specimens 1 and 3 to isolate the effects of the leaching agent, if any.

Specimen 3 was the control specimen to determine if the use of MEK in specimens 1 and 2 caused any variation in the spectrum due to absorbance, adsorbance, or chemical reaction, and to prove that this method is feasible.

Since specimen 1 was pure epoxy and had no glass in it, all that was necessary to make a spectrum of it was to grind it in a mortar, mix it with potassium bromide (KBr), and prepare a KBr wafer. KBr was chosen for the wafers because it is transparent from 2.50 microns to 25 microns and has no bands of adsorbance in the infrared range.

Specimens 2 and 3 were treated differently because it is necessary to separate the fiberglass from the epoxy composite. The differential flotation method of separation was used, employing the concept of floating one material from a mixture of two different materials having different specific gravities in a liquid of intermediate specific gravity. In this case the epoxy, with a specific gravity of 1.25, can be floated from the fiberglass, with a specific gravity of 2.15, by washing the ground mixture in methylene chloride (CH_2Cl_2), which has a specific

gravity of 1.336. The liquid (CH_2Cl_2) does not react with either the cured epoxy or the glass.

Approximately a 2-1/2-gram ground sample was obtained from each of specimens 2 and 3 by filing the specimens with a double-cut bastard file. These two samples were thoroughly washed with CH_2Cl_2 and centrifuged at least three times each, discarding the fiberglass precipitate after each centrifugation. The resulting flotation or emulsion was allowed to evaporate to dryness at 40°C, leaving the pure ground epoxy as residue. Potassium bromide was ground and mixed with each specimen, and KBr wafers were made as with the pure epoxy, specimen 1.

The wafers were put into the sample holder of the spectrophotometer and placed in the desiccator; the holder was positioned in the sample beam, and the spectrophotometer was activated. The spectrograms shown in Figures 1, 2, and 3 were obtained. Comparison of these three figures shows that the spectrograms are alike, proving the feasibility of this approach.

CONCLUSION

Based on the results of this evaluation, it is concluded that a true infrared spectrum of a cured epoxy can be obtained by this method; thus, the components can be accurately analyzed.

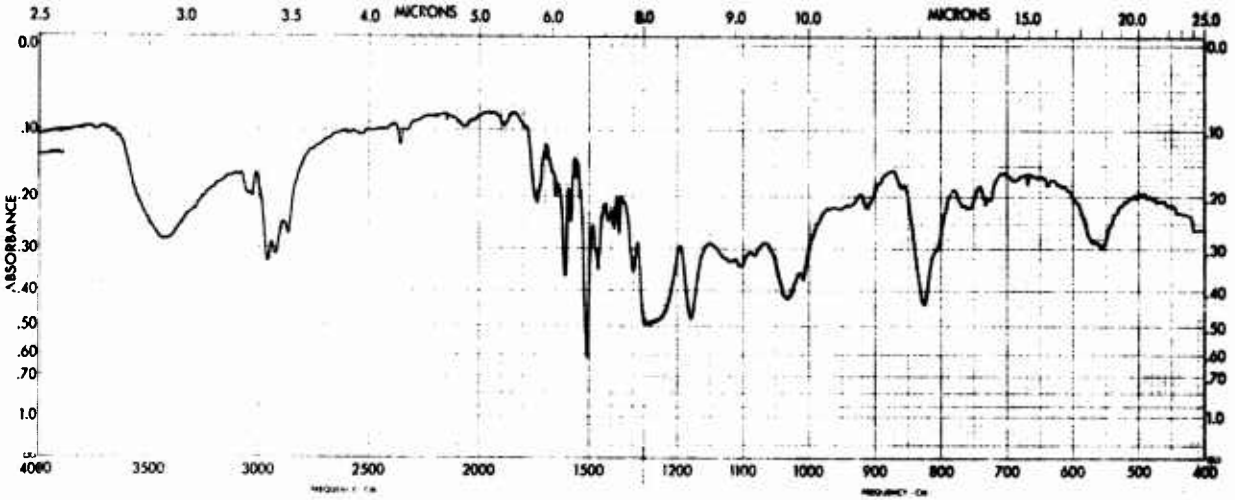

Figure 1. Specimen 1 - Pure Epoxy Leached With Methyl Ethyl Ketone.

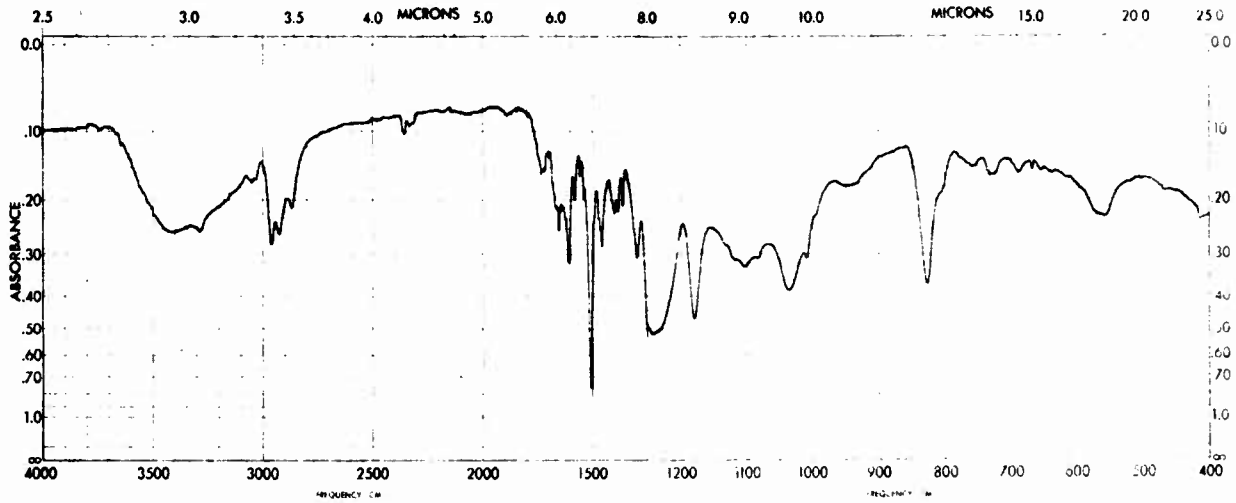

Figure 2. Specimen 2 - Epoxy Plus Glass Leached With Methyl Ethyl Ketone.

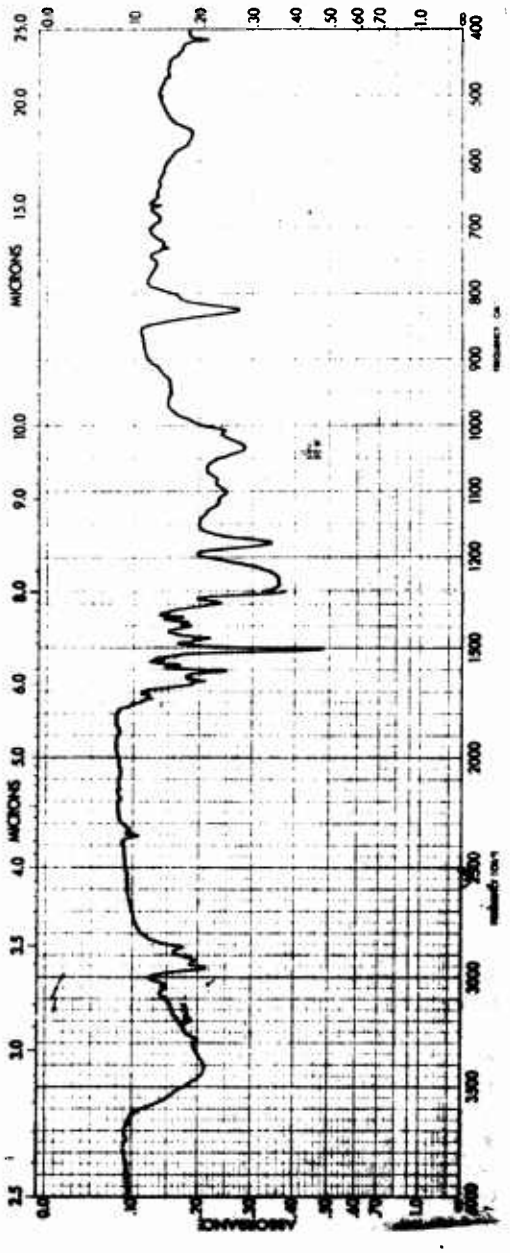

Figure 3. Specimen 3 - Epoxy Plus Glass Not Leached With Methyl Ethyl Ketone.

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