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FTIR Spectral Evaluation of Polyurethane Adhesive Bonds in Perspex Canopies of Aircraft

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ABSTRACT

Failure of capron glue (polyurethane) adhesive joint between nylon strips and perspex canopy of aircraft has been studied by Fourier transform infrared spectral analysis. It is concluded that the brittle failure of the joint is due to time-dependent crosslinking of residual isocyanate group with residual hydroxyl or the amino groups produced as a result of reaction of absorbed moisture with the isocyanate moiety. A service life of eight years has been estimated for the glue joint.

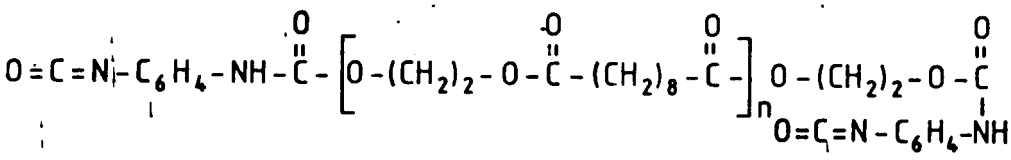
1. INTRODUCTION

Fourier transform infrared (FTIR) spectral analysis of the ageing characteristic of the polyurethane capron glue joint between nylon strips and perspex canopies of aircraft has proved to be a useful tool for prediction of failure. The gradual disappearance of the isocyanate absorption at 2350 cm^{-1} - 2360 cm^{-1} indicated progressive curing of the residual isocyanate groups over several years of use leading to brittle failure of the adhesive. In the recent past, failure of the bond between nylon strips holding the perspex canopies in position in a few aircraft leading to accidents in air had been suspected.

Therefore, an investigation of the failure of the capron glue joint by some suitable spectral method, which could provide continuous recording of data on the joint material that had failed, was considered worthwhile. It was found that the adhesive used for joining the nylon strips with perspex canopies of the aircraft is a polyurethane condensate of sebacic acid-ethyleneglycol diol ($\overline{M}n$ = 2000) and toluene diisocyanate. As the cured material did not dissolve in any solvent and the *KBr* pellet of the crosslinked adhesive did not give any sharply resolved peak in the IR spectrum, it was decided to go for the FTIR spectral analysis of the *KBr* pellets of the material. Interestingly, this resulted in finding of a method to predict the failure of the adhesive bond, with useful accuracy.

2. EXPERIMENTAL METHOD

A total of 222 samples of small strips of perspex cut from the canopies of the aircraft joined with nylon strips with adhesive of varying period of ageing starting from 7 to 13 years, were procured. A fresh sample which had not aged was also procured. Almost all the samples were tested for the ultimate mechanical strength. The joint material was then scrapped out of the nylon strips carefully without affecting the nylon beneath. The



Structure 1: Polyurethane (capron glue) adhesive molecule.

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powdered material thus obtained was used to make *KBr* pellets for FTIR analysis. The FTIR spectrophotometer used was a Perkin Elmer Model 1605, computerised equipment.

3. RESULTS & DISCUSSION

From the large number of samples, a random choice of three samples was made from each lot belonging to a particular year of ageing of the joint material for FTIR spectral analysis. Samples with 7 to 8 years of ageing had a strong absorption peak of 2350 cm^{-1} - 2360 cm^{-1}, assigned to the assymetric stretching of the isocyanate group (*-N=C=O*). To get rid of the problem created in the peak heights due to varying amount of test material in the *KBr* pellets, the carbonyl (*>C=O*) peak at 1620 cm^{-1} - 1660 cm^{-1} was chosen to be the standard peak to produce a ratio of the peak heights at 2350 cm^{-1} - 2360 cm^{-1} and 1620 cm^{-1} - 1660 cm^{-1} in each sample for comparison. Table 1 provides the comparison of isocyanate peak absorption divided by the carbonyl (*>C=O*) peak absorption averaged over three samples in each case for different years of ageing.

Table 1. FTIR analysis data for aged capron glue adhesive of the aircraft canopies

S. No.	Years of ageing	Samples	Peak height h_1 (cm)	Peak height h_2 (cm) at 2350 to 2360 cm^{-1}	Peak height at 1620 to 1660 cm^{-1}	h_1/h_2	Mean h_1/h_2
		1	14.6	2.15	6.34		6.67
		2	16.00	2.15	7		
		1	12.8	2.6	4.92		
		2	5.75	1.2	4.6		4.76
		1	7.7	2.5	3.08		
		2	3.6	1.2	3.00		3.13
		3	6.3	1.9	3.315		
	10	1	5.2	2	2.6		
			5.00	2.2	2.2727		2.89
			7.8	1.9	3.85		
	11	1	1.2	1.1	1.0909		
		2	1.5	1.00	1.5		1.295
6*	12		0.6	3.1	0.1909		0.1995
7*	13		nil	nil	nil		nil

h_1 = Peak height for isocyanate group absorption
h_2 = Peak height for carbonyl roup absorption
* Received two samples only

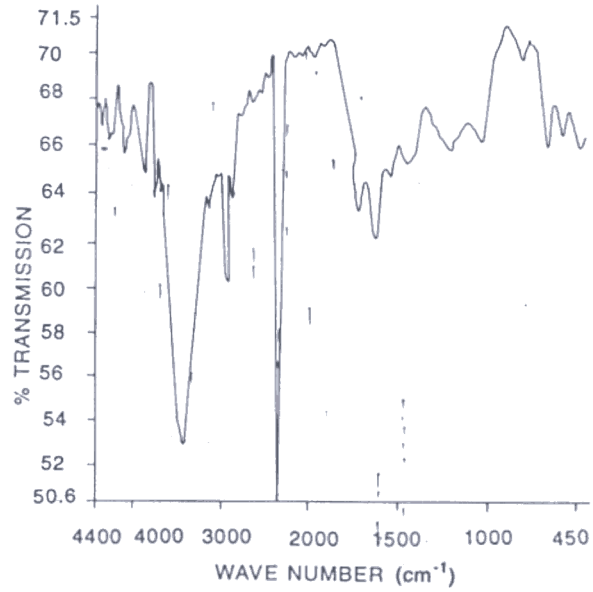

Figure 1. FTIR spectrum of a sample of cured polyurethane capron glue aged for 7 years which did not fail.

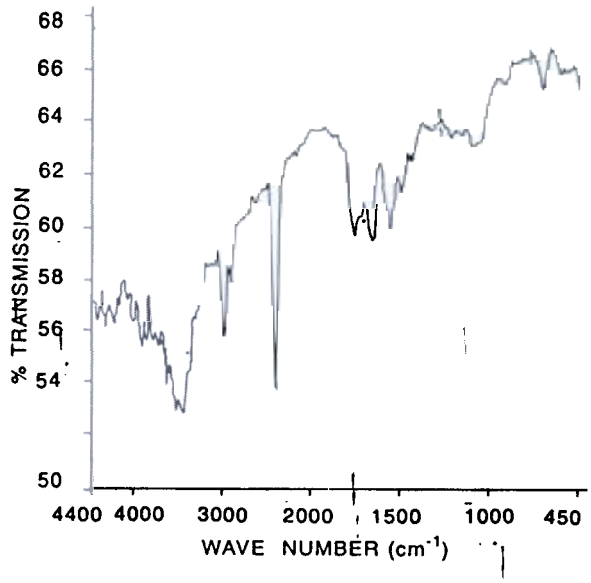

Figure 2. FTIR spectrum of a sample of cured polyurethane capron glue aged for 10 years which failed.

Figure 1 gives FTIR spectral of a sample of polyurethane capron glue aged for seven years, which did not fail. This was compared with the spectrum of a similar sample with reduced isocyanate absorption that failed after 10 years ageing as given in Fig. 2.

When the results reported in Table 1 were put into a graph as shown in Fig. 3, after nine years of ageing a smoothly decreasing curve showed irregular deviations. This can be attributed to the brittle fracture

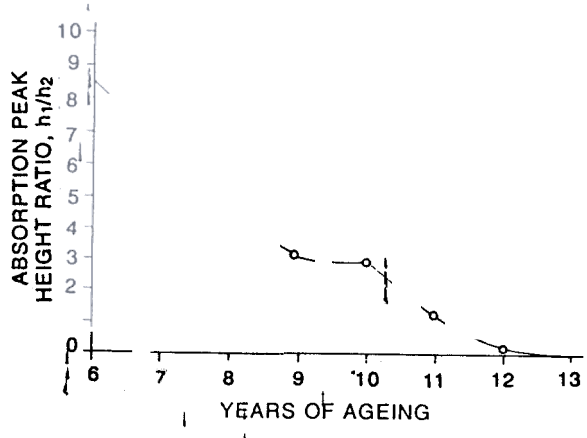

Figure 3. Ratios of peak heights, h_1/h_2 versus years of ageing for cured polyurethane capron glue.

in the adhesive joints, as most of the joints failed after 9 years of continuous ageing. Based on these results, it is recommended that the adhesive joints be replaced by fresh joints after 8 years of continuous service, when the ratio of absorption peak height of the isocyanate group and that of the carbonyl group is around 4.76. Interestingly, this recommendation falls in line with those of the manufacturers of aircraft for the replacement of the aged joints between nylon strips and perpex canopies. These results also help to know the number of years of use of an aircraft by FTIR spectral testing of the polyurethane adhesive used for bonding the aircraft canopies and the nylon strip holders.

As the samples of the polyurethane glue with 1 to 6 years of ageing were not available from the aircraft that remained in active service, the present work leaves scope for further investigation. However, with the available FTIR data, a chemical explanation for the brittle failure of the glue joint between nylon strips and perpex canopy can be attempted. FTIR spectra of all the samples tested possessed strong absorption for *-OH* groups in between 3500 cm^{-1} to 3000 cm^{-1}, in addition to absorption for residual (*-N=C=O*). The *-OH* groups may belong to the hydroxy-terminated polyester that has not or only partially reacted with (-N=C=O) group of toluene diisocyanate. The *-OH* group may also come from absorbed moisture over long years of outdoor exposure. It is well-known that the reaction of moisture with isocyanate group produces CO_2 and $-NH_2$ group with evolution of heat. Both *-OH* and $-NH_2$ groups, thus available, are known to enter into crosslinking reaction with the isocyanate group of toluene diisocyanate[1]. This leads to an increase in crosslinking density with a fall in h_1/h_2 (peak height for isocyanate groups absorption/peak height for carbonyl group absorption) till a thershold value is obtained when the joint turns sufficiently brittle. The horizontal portion of the curve in Fig. 3 may refer to this threshold limit of h_1/h_2. After failure of the joint, the residual (*-N=C=O*) groups of the fresh surface react with atmospheric moisture resulting in a sharp fall of h_1/h_2. The magnitude of h_2 is not affected as much as that of h_1 as the carbonyl groups occur in the ester moiety in addition to being a part of the isocyanate group.

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