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NOTE

Some Properties of Glues Based on Interpenetrating Polymeric Networks

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Preparing interpenetrating polymeric networks (IPN) is useful as a basically novel method of mixing non-melting and insoluble three-dimensional polymers. IPN are known to be characterized by a number of thermodynamical and physico-mechanical properties.¹ Preparing IPN on the basis of cross-linked polymers of different composition yields materials with a broad range of properties, but employment of them as glues has not been reported in the literature.

We have for the first time established basic peculiarities of IPN used as adhesives, arising from differences in curing rates of the two networks in an IPN. These peculiarities will be discussed on the example of glues based on IPN, consisting of polyester (PE) and polyurethane (PU). The IPN were obtained by the method based on simultaneous formation of two networks. A solution of oligodiethyleneglycolmaleinatephthalate (PE-resin) in styrene was mixed with macrodiisocyanate (MDI)—a product of interaction of 1 mole of oligodiethyleneglycoladipate and 2 mole of toluylenediisocyanate and cured under the action of 2% methyl ethyl ketone peroxide and 1% cobalt naphthenate. The end hydroxyl and carboxyl groups of the PE resin were, prior to this, bonded by phenol isocyanate. The resin cured in 40 minutes.

Polymerization of the MDI occurred due to the interaction of the isocyanate groups with moisture of the air that diffused into the glue joint for 90 to 110 days. This was established by IR spectroscopy on the band of valency

oscillations of the isocyanate groups at 2280 cm^{-1} . Copolymerization of the PE resin with the MDI with further formation of the substituted amines, whose probable occurrence had been indicated in Ref. 2, was not observed in the present system. Ratio of PE to MDI was 1:0.8. Two states of the polymer in the glue joint can be distinguished because of slow polymerization of the MDI. At the first stage immediately after polymerization of PE resin the glue is a PE plasticized by the MDI. At the second stage the glue is an IPN formed after polymerization of the MDI.

With the progress of IPN formation properties of the system change—modulus of elasticity (Table I) and adhesive strength (Figure 1) increase.

TABLE I
Variation of the modulus of elasticity of the IPN glue joint vs. time

Time after curing, days	Modulus of elasticity, kg/cm^2	
	Torsion displacement	Elongation displacement
5	4.0×10^2	4.2×10^2
100	2.4×10^3	0.1×10^3

During the first day after curing, failure of the adhesive joints is of the adhesive nature and after 5 to 10 days it becomes cohesive—in the adhesive layer.

Figure 1 shows that adhesive strength of the IPN considerably surpasses that of either PE or PU used as glues. One of the reasons for this is the low level of internal stresses in the glue joint of the IPN as compared with the PE (Figure 2) which can be explained by the increased rate of relaxation of internal stresses in the glue joint because of the presence of a considerable amount of liquid MDI.

As polymerization of the MDI took place for a long period of time the stresses relaxed almost completely; even 100 days later internal stresses did not exceed $10\text{--}15\text{ kg/cm}^2$. Internal stresses in the glue joints were determined from the curving of the fixed three-layer bar.³ The metals glued were steel and aluminium.

As is known⁴ reduction of internal stresses in the glue joints causes increase of the adhesive strength under continuous load. In employing IPN of this type as glues the strength under continuous load exceeds that under instantaneous load with loads up to 55% of ultimate (Figure 3). Increase of strength under continuous and instantaneous load takes place in approximately the same period of time, and roughly the same time is required for isocyanate groups to react (cf. Figures 1 and 3).

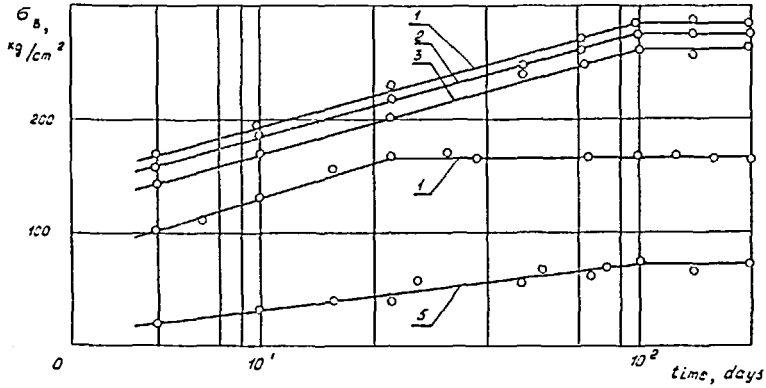


FIGURE 1 Variation of the adhesive strength of the glue joints vs. time. The materials and glues: 1—steel IX13HIOT; 2, 4, 5—steel st.3; 3—alloy D-16; 1-3—glue on the basis of IPN; 4—PE glue; 5—PU glue.

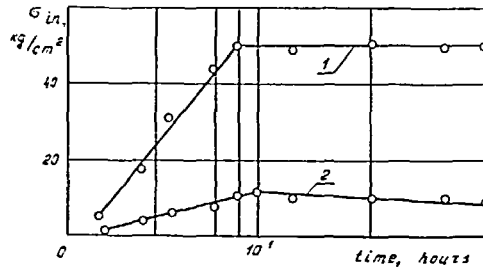


FIGURE 2 Increase and relaxation kinetics of the internal stresses vs. time. 1—PE glue; 2—glue on the basis of IPN.

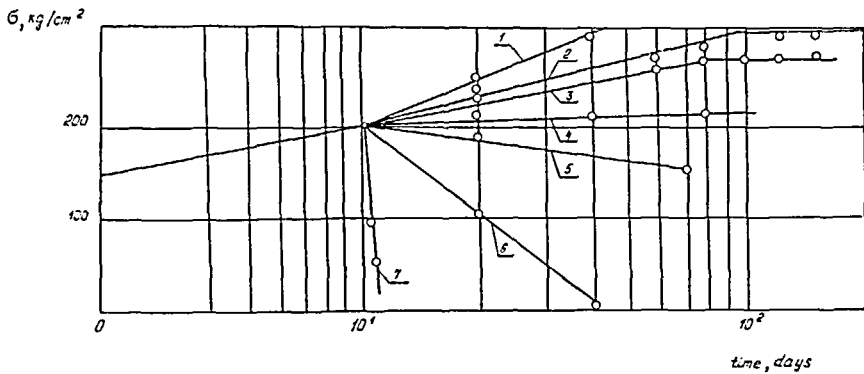


FIGURE 3 Strength of the IPN glue joints under continuous load (curves 1-5 and 7); and PE glue joints (curve 6). The joints were loaded 10 days after glueing. Load level: 1—0.3; 2 and 6—0.5; 3—unloaded; 4—0.55; 5—0.6; 7—0.8.

This evidence testifies to the fact that increase of strength of the glue is connected with IPN formation processes. Now we shall deal with the effect of load application on these formation processes.

Under the action of load the initially elastic glue joint is deformed. As the network deforms, MDI molecules present in its interstructural regions orient corresponding to the vector field, changing their conformation, which should increase the cohesive and adhesive strength. This concept is supported by the following experiments. The glue joints were subjected to normal or tangential stresses and then tested at different ratio of normal and tangential stresses. Strengthening of the glue joint was observed mainly in the direction parallel to that of the previously applied load.

Tests of the adhesive strength were performed with instruments and according to the technique described in Ref. 5. Figure 4 shows that glue joints, subject for 100 days to the action of shear (curve 2) or normal (curve 3) load equal to 30% of the breaking load, had increased resistance to shear and normal loads.

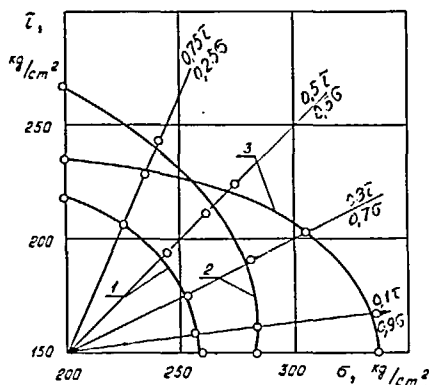


FIGURE 4 Strength of the glue joints with different ratio of normal and tangential stresses 100 days after glueing; 1—the specimens were unloaded; 2—displacement load—0.3; 3—normal load—0.3.

Considerable change in conformation of macromolecules was observed if polymerization took place within the shear stress gradient.⁶ In this case adhesive strength increased by more than 50%, mostly along the stress direction.

Polymerization of the glue was studied when it was pumped through a narrow gap between the glued surfaces. If the pumping was terminated before fluidity was lost, adhesive strength would either not increase at all, or the percentage increase was much lower. In this case, macromolecules evidently have time to assume initial conformation.

Insignificant increase of the adhesive strength of the glued specimens, in the direction perpendicular to the action of continuous load, can be explained by "healing" flows by MDI in the polymeric structure that develops in the loaded glue joint. Thus, the basic peculiarity of employing glues based on IPN lies in essential decrease of the level of internal stresses in the glue joints, with simultaneous increase of adhesive strength under instantaneous and, particularly, continuous load. These peculiarities are determined by difference in curing kinetics of the two networks and resulting specific conditions of relaxation of the internal stresses, markedly affecting adhesive strength.

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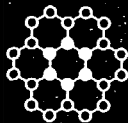
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