Microstructures of unsaturated polyester resins modified with reactive liquid rubbers[*]

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Abstract: The work presents an analysis of the microstructure of polyester resin castings modified with two reactive liquid rubbers, which were dissolved in the resin and precipitated during curing. Samples of castings were imaged with an optical and a scanning electron microscope. The volume fraction of precipitated particles is 3–10 times greater than the introduced volume of rubber. The particles contain not only rubber, but also (co)polymerization products of styrene and/or polyester and show good adhesion to the polyester resin matrix. The size of the particles is dependent on the type and amount of rubber used.

Keyword: unsaturated polyester resin, resin modification, reactive liquid rubber, microstructure, morphology.

Mikrostruktura nienasyconej żywicy poliestrowej modyfikowanej ciekłymi kauczukami reaktywnymi

Streszczenie: Analizowano mikrostrukturę odlewów żywicy poliestrowej modyfikowanej dwoma rodzajami ciekłych kauczuków reaktywnych, które rozpuszczały się w żywicy i wytrącały podczas sieciowania. Odlewy obserwowano przy użyciu mikroskopów optycznego i elektronowego. Udział objętościowy wytrąconych cząstek był 3–10 razy większy niż objętość kauczuku wprowadzonego do osnowy poliestrowej. Wydzielone cząstki zawierające nie tylko kauczuk, ale również produkty polimeryzacji styrenu i/lub poliestru lub produkty kopolimeryzacji styrenu z poliestrem wykazywały dobrą adhezję do osnowy. Rozmiar wytrącających się cząstek zależał od rodzaju użytego kauczuku i jego zawartości w żywicy poliestrowej.

Słowa kluczowe: nienasycona żywica poliestrowa, modyfikacja żywicy, ciekłe kauczuki reaktywne, mikrostruktura, morfologia.

Polymer composites, especially fiber-reinforced plastics (FRP) are nowadays ever more common as construction materials in aerospace, motorization, civil, nautical and wind-power constructions. In these areas of use, FRP are subjected to various loads, among which one can find impact loads.

Thermosetting resins are commonly used as a polymer matrix in FRP: unsaturated polyester (UP), vinyl ester (VE), epoxy and phenolic resins. These are, upon curing, brittle solids. This brittleness is unfortunate in FRP applications because it provokes a brittle failure of the composite, especially under impact loading.

Since the 1960s, construction-grade UP resins are often blended with flexible UP resins to enhance their impact strength, as well as other properties. Their cross-link density is far lower than in construction grade orthophthalic resins; thus, the elasticity and toughness are increased, which is desirable. Unfortunately, at the same time, hardness, elastic modulus, compression strength and thermal resistance are decreased [1–3].

Since the 1970s, additions of reactive liquid rubbers (RLR), also called reactive liquid polymers (RLP), are used to improve the toughness and impact strength of epoxy resins, *e.g.*, [4–8]. Nowadays, it is a common method for enhancing the performance of epoxy resins. This success led to trials of RLR for the modification of other types of resins: phenolic [9] as well as vinyl ester and unsaturated polyesters. Trials with the latter resins were first communicated in the 1980s, *e.g.*, [10–12]. Good reviews of this subject are found in [13, 14].

There are a few methods of UP or VE resin modifications with RLR. Among them, the first, and probably the

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one with widest use consists of dissolution of the reactive liquid rubber in the liquid resin and subsequent curing [10–17]. For the unsaturated polyester resins, liquid rubbers with vinyl or epoxy end-groups are used because of their greater compatibility [10–16]. These rubbers should dissolve in the liquid resin and precipitate during the curing reaction, forming a fine dispersion of rubber particles – the size of these particles has a strong effect on the mechanical properties of the composition [10–12]. The second frequently mentioned method is block copolymerization of unsaturated polyesters and rubbers [17, 18].

The RLR modification of UP and VE resins is used to increase resin toughness, *i.e.*, resistance to brittle failure [10–18]. This improvement may be a result of one of several mechanisms, *e.g.*, cavitation and bridging, among others [14]. This effect should gradually disappear as the loading rate increases, with no expected increase in impact strength [11, 19].

This contribution is a part of a wider work on toughened glass-fiber/polyester composites. Previously published data were: results of rheological-processing tests of RLR modified UP resin [20], mechanical properties of cured casts of RLR modified UP resin [21, 22], and mechanical properties of composites produced using the RLR modified UP resin [23–25].

EXPERIMENTAL PART

Materials

– The basic resin used in this investigation was Polimal 109-32K unsaturated polyester resin manufactured by Z.Ch. Organika-Sarzyna (Poland). It is a standard, rigid, high-hardness, medium-reactivity, low-viscosity orthophthalic resin with styrene as a vinyl monomer.

– This resin was modified with the following two reactive liquid polymers:

Hypro™ ETBN 1300x40 – epoxy-terminated butadiene-acrylonitrile polymer dissolved in styrene (50 %), and Hypro™ VTBNX 1300x33 – vinyl-terminated butadiene-acrylonitrile reactive liquid polymer; both RLPs were manufactured by Emerald Performance Materials (USA) and supplied by Nanoresins AG (Germany). The properties of the RLPs are presented in Table 1.

The ETBN, which was a premix in styrene, was mixed directly with the resin. The VTBNX was either mixed directly with the resin or mixed with styrene (50 %) first. The resultant, low-viscosity liquid was then mixed with the resin.

– The curing system in all cases consisted of 0.6 cm^3/kg of 10 % cobalt accelerator (ILT, Poland) and 1.5 wt % of Metox 50 initiator (methyl ethyl ketone peroxide solution manufactured by Oxytop Ltd., Poland).

Sample preparation

Compositions with the following additions were prepared for use in this study: 2 phr ETBN premix (1 phr rubber), 6 phr ETBN premix (3 phr rubber), 10 phr ETBN premix (5 phr rubber), 1 phr neat VTBNX, 2 phr VTBNX premix (1 phr rubber), 6 phr VTBNX premix (3 phr rubber), 10 phr VTBNX premix (5 phr rubber).

T a b l e 1. Properties of selected Hypro™ reactive liquid polymers [26, 27]

Hypro™ polymers	VTBNX 1300x33	ETBN 1300x40
Bound acrylonitrile, %	18	8.5
Molecular weight	3 880	–
Acid number	0–5	1.5 (max.)
Functionality	2.2	1.8
Brookfield viscosity at 25 °C, mPa · s	250 000	1 450
Density, kg/m^3	964	945
Specific gravity at 25 °C	0.967	0.945
Solubility parameter [a]	8.898	N/A[c]
Glass-transition temperature, °C [b]	-49	N/A[c]
Total solids, %	100	50 ± 2

[a] Calculations based on molar attraction constants.

[b] Measured *via* DSC.

[c] Not available.

The compositions were cured with the curing system at ambient temperature in a mold for 24 hours, then demolded and post-cured, also at ambient temperature, for 30 days.

Methods of testing

– Sections of the cured compositions were examined under an optical microscope. Prior to examination, the samples were ground and polished with abrasive materials of gradually decreasing grain size. The microscope used was a Carl-Zeiss-Jena Jenavert equipped with a Panasonic Color CCTV Camera Model WV-CD132L for image capture. Digital image analyses were performed upon the obtained images in order to estimate (quantitatively) the volume of the precipitated rubber particles. The estimation was performed according to the usual guidelines [28]. The software used for the analysis was Scion Image. The images were thresholded (the threshold level was governed manually to obtain the closest approximation of visually-discerned particles), and the area of the particles was counted automatically.

– Fracture surfaces of the cured compositions were examined under a scanning electron microscope (SEM). Samples were fractured in liquid nitrogen, mounted on a stand and sputtered with gold to prevent a build-up of electrical charge. The electron microscope used was a Jeol JSM-6100.

– In order to analyze the nature of the particles in the cured compositions further, microhardness (HV) tests were performed on the particles. The microhardness tester used was a Clemex CMT-HD. The indenter was a typical Vickers' pyramid-shaped diamond with 136°

apex angle. This test was performed only upon the 10 phr VTBNX-premix material due to the insufficient size of the particles in the other materials.

– For the purpose of phase-composition analyses, differential scanning calorimetry (DSC) tests were performed upon samples cut from the castings. Only materials with the highest rubber content were tested due to their highest possible signal-to-noise ratio. The tests were carried out on a TA Instruments Q-Series Q100 instrument. Samples were heated from -80 to 250 °C at a constant rate of 10 deg/min in an inert atmosphere. From the DSC curve, glass-transition temperatures were estimated.

RESULTS AND DISCUSSION

Figures 1–4 present optical micrographs of the morphology of samples of the compositions with varying ETBN and VTBNX content. It was seen that precipitated particle size increased with higher RLP contents. In the case of the composition with VTBNX mixed directly with resin, there were visible agglomerations of the rubber phase (Fig. 1a and 3a). These were not observed in the compositions where VTBNX or ETBN were first premixed with styrene. Therefore, direct mixing of very viscous RLP with low-viscosity resin may be insufficient to produce an effective mixture. The particle size in the VTBNX-modified resin was visibly larger than in the ETBN-modified resin. In compositions with 10 phr VTBNX premix (Fig. 1d and 3d), the typical size of most of the visible particles in the VTBNX-modified resin was in the range of 30–100 μm, while in the ETBN-modified resin (Fig. 2c and 4c) it was in the range of 10–30 μm. Larger particles displayed internal differentiation (see esp. Fig. 4c).

Table 2 presents the estimated volume fraction of precipitated, presumably rubber particles in each composition. Strikingly, the volume fractions are 3–10 times higher than the introduced RLP level. The increased volume fraction compared to the added volume of rubber have been noted previously by Robinette *et al.* [15] and Ullet and Chartoff [16] but was not further analyzed by the latter. It is evident that the precipitated particles contain some other substance besides rubber. The most probable candidate is styrene from the UP resin – it has low molec-

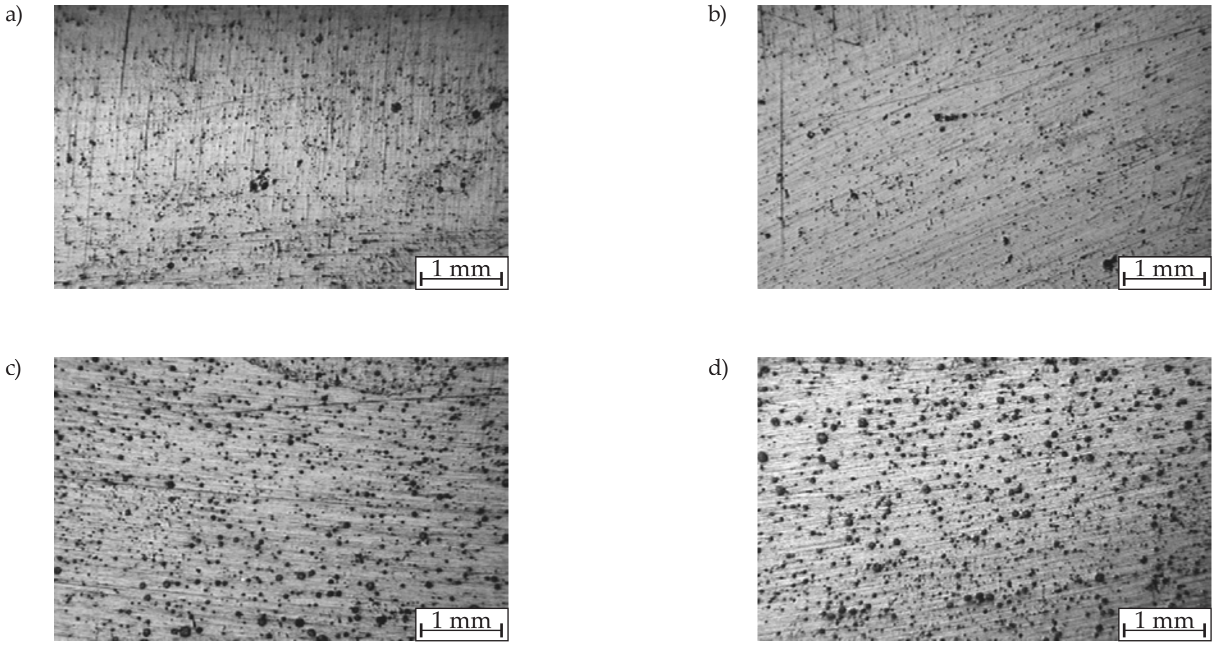

Fig. 1. Optical micrograph of compositions modified with VTBNX (magnification 64x): a) 1 phr rubber, b) 2 phr premix, c) 6 phr premix, d) 10 phr premix

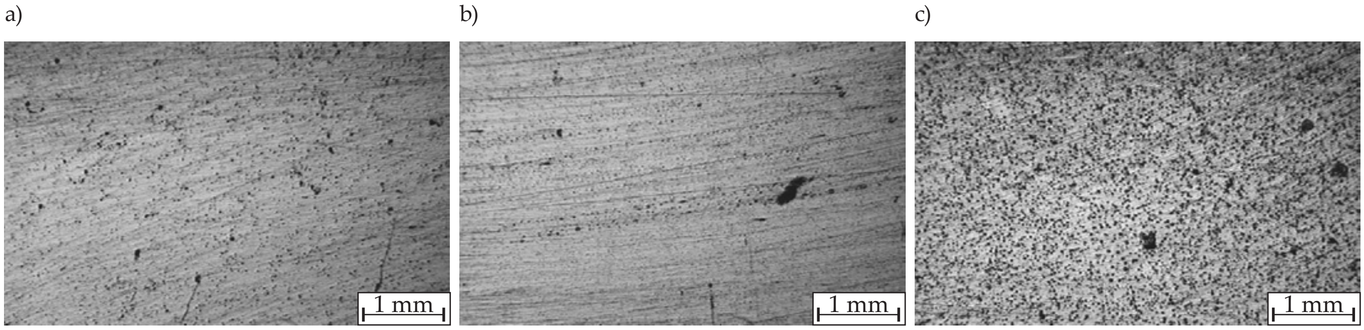

Fig. 2. Optical micrograph of compositions modified with ETBN (magnification 64x): a) 2 phr premix, b) 6 phr premix, c) 10 phr premix

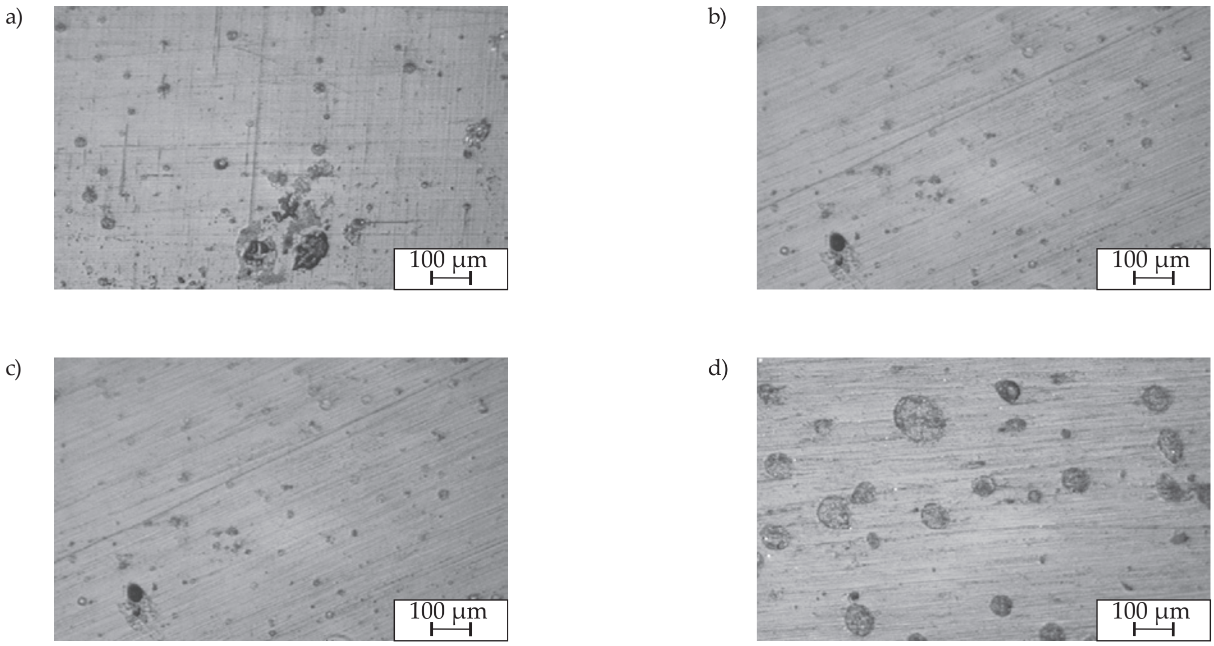

Fig. 3. Optical micrograph of compositions modified with VTBNX (magnification 320x): a) 1 phr rubber, b) 2 phr premix, c) 6 phr premix, d) 10 phr premix

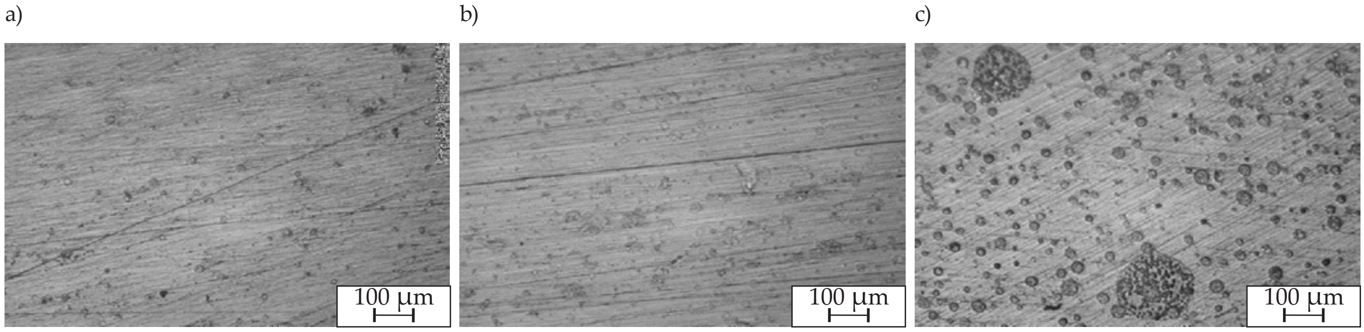

Fig. 4. Optical micrograph of compositions modified with ETBN (magnification 320x): a) 2 phr premix, b) 6 phr premix, c) 10 phr premix

ular weight (therefore migrates easily) and is completely miscible with the RLP. Styrene migration – although in exactly the opposite direction – during curing was proposed by Robinette *et al.* [15]. A significant addition of some substance (or substances – there may be more than one) besides rubber was also evidenced by the results of DSC and microhardness tests. The latter revealed the hardness of particles in 10 phr VTBNX premix modified resin in the range of 15–16 HV (compared to 27 ± 1 HV of the resin matrix), which is comparable to the hardness of acrylonitrile-butadiene-styrene (ABS) terpolymer [29] and much above the hardness of nitrile-butadiene rubbers.

T a b l e 2. Estimated volume fractions of precipitated phase in each composition

Composition	Rubber content		Precipitated phase volume ratio vol %
	wt %	vol %	
1 phr neat VTBNX	1.0	1.3	8.9
2 phr VTBNX premix	1.0	1.3	11.2
6 phr VTBNX premix	2.8	3.5	15.5
10 phr VTBNX premix	4.5	5.6	18.4
10 phr ETBN premix	4.5	5.7	22.8

The DSC revealed (Fig. 5), besides a resin glass-transition at a temperature around 75 °C, a second glass-transition at a temperature around -15 °C, which fits neither the resin nor rubber (typical or RLP; Hypro™ VTBNX 1300x33 glass-transition temperature is -49 °C [26, 27]). It is evident that the cured compositions contain at least one phase different from either resin matrix or rubber. The glass-transition temperature suggested that this phase contains more than 50 % rubber in styrene. At the same time, microhardness measurements suggested the presence of a phase containing a significantly higher level of styrene, besides the rubber. Such a phase would have a glass-transition temperature very similar to that of resin and thus masked. To evaluate the constitution of these rubber-containing phases would require the use of high-resolution microscopic ATR FTIR (attenuated total reflectance Fourier transform infrared spectroscopy) or Raman spectra measurement.

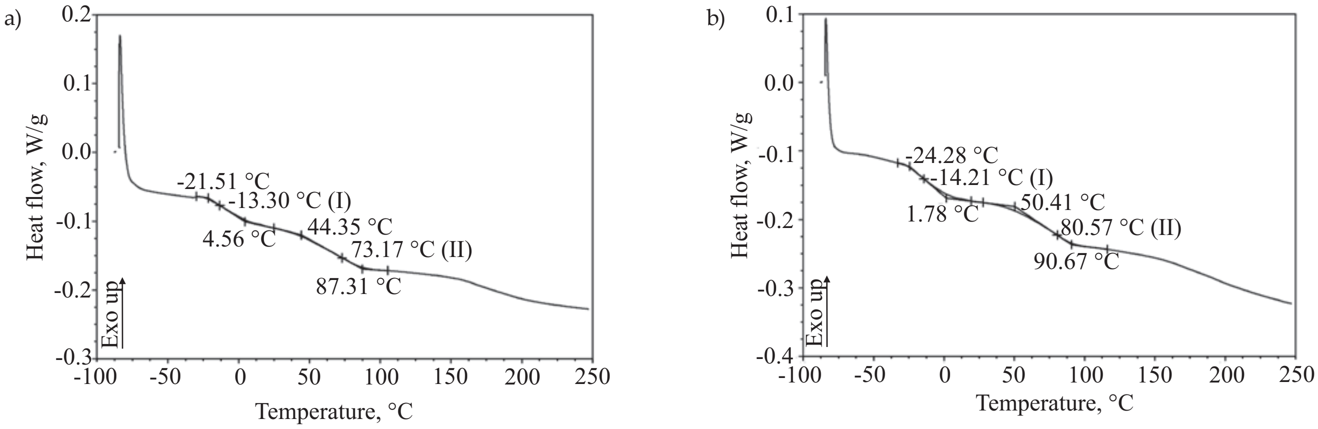

Fig. 5. DSC results for compositions: a) 10 phr VTBNX premix, b) 10 phr ETBN premix

Figures 6–12 present SEM images of the precipitated rubber-containing particles in the compositions with varying ETBN and VTBNX content. It may again be seen that the size of particles is larger in VTBNX-modified resin than in the ETBN-modified one. With increasing rubber content, the particles size increases. In the VTBNX-modified resin, the particles are hard to distinguish from the matrix (Figs. 6–9). Cracks propagate through the particles, rather than around them. The particles are visible mainly due to the greater smoothness of their fracture surface and darker shade of grey. In compositions with higher concentrations of rubber (Fig. 9), the level of the surface seems to be stepped compared to the level of the matrix surface. This is not observed in lower-concentration compositions. Particle borders are well defined (not fuzzy) but difficult to discern. No gap between phases is visible, positing perfect adherence. The particles in VTBNX-modified resin exhibit internal differentiation – in the primary particles,

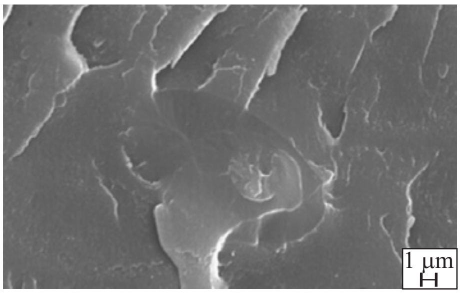

Fig. 6. SEM image of composition with 1 phr neat VTBNX

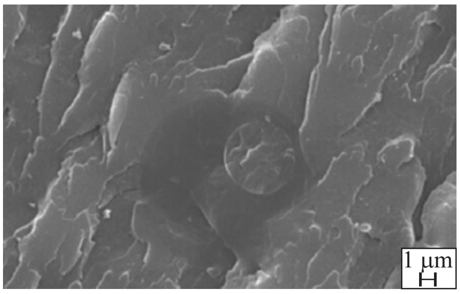

Fig. 7. SEM image of composition with 2 phr VTBNX premix

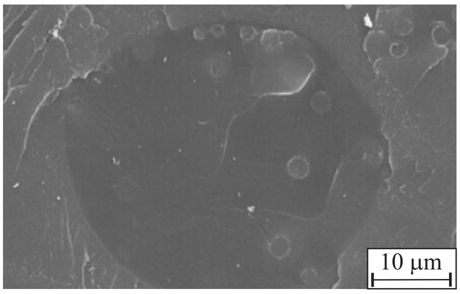

Fig. 8. SEM image of composition with 6 phr VTBNX premix

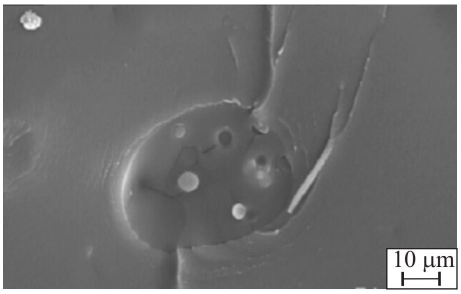

Fig. 9. SEM image of composition with 10 phr VTBNX premix

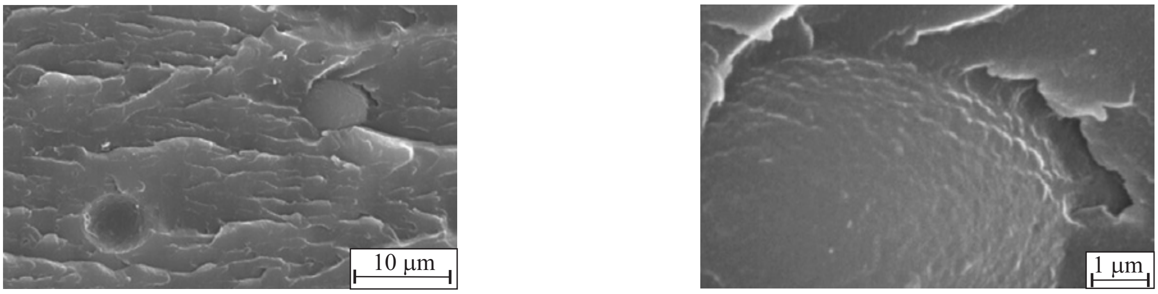

Fig. 10. SEM images of composition with 2 phr ETBN premix

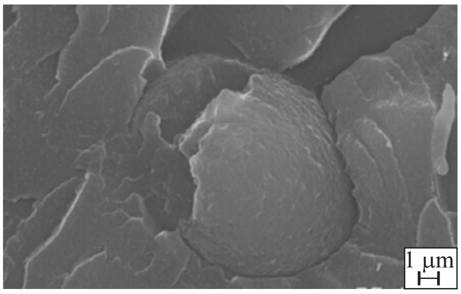

Fig. 11. SEM image of composition with 6 phr ETBN premix

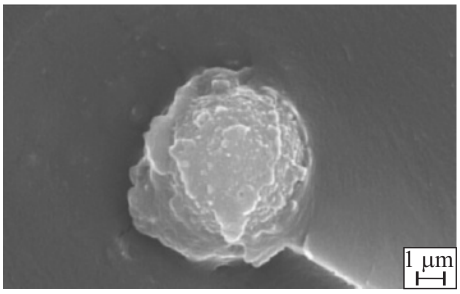

Fig. 12. SEM image of composition with 10 phr ETBN premix

secondary precipitated domains are visible. This internal differentiation becomes more pronounced with increasing rubber content; the secondary domains become larger in number and size (Figs. 8, 9). It may be hypothesized that the precipitated particles contain not only rubber but also styrene. The heterogenic particles would, therefore, consist of a rubber-rich phase domain enclosed in a styrene-rich phase. Cracks propagate through the styrene-rich phase and either around or through the rubber-rich phases. The postulated particle structure is illustrated in Fig. 13. The internal differentiation of particles in VTBNX-modified (and, speculatively, ETBN-modified) VE resin was postulated, but not confirmed, previously by Robinette *et al.* [15].

There is no visible difference in structure between compositions with 1 phr neat VTBNX (Fig. 6) and 2 phr VTBNX premix (Fig. 7) under SEM.

In ETBN-modified resin (Figs. 10–12), the particles are generally smaller than in VTBNX-modified ones (contrary to the findings of Robinette *et al.* [15]). There are different sizes of particles at every concentration, without a clear increase in size visible with higher rubber contents. The particles are *quasi*-spherical with a rough surface. In the majority of cases, cracks propagate around the particles without penetrating them; the particles are visible intact, or impressions of them are left. In a few cases, the particles are also fractured (Fig. 11). Nevertheless, the adhesion between the particles and the matrix is good, as evidenced by fuzzy borders between phases (see esp. Fig. 10 right). This is contrary to the findings of Robinette *et al.* [15], where cavitated gaps around ETBN particles were present in vinyl ester matrix. It is impossible to tell whether the particles are internally differentiated because most particles are intact and no internal structure is visible.

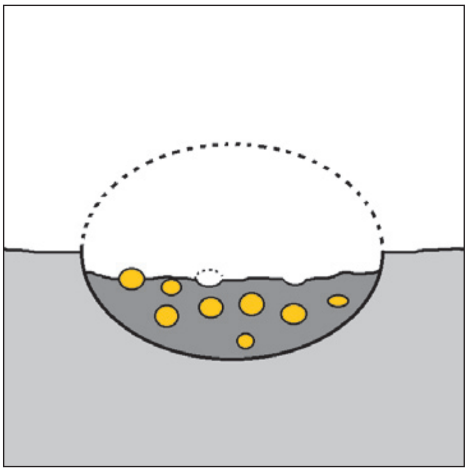

Fig. 13. Postulated structure of precipitated particle in VTBNX-modified resin

CONCLUSIONS

In this study, compositions of orthophthalic, unsaturated, polyester resin modified with two reactive liquid rubbers – vinyl- and epoxy-terminated butadiene-acrylonitrile rubbers – were prepared with varying rubber contents. These compositions were cured and their morphology was inspected using optical and scanning electron microscopy. Additional tests of DSC and microhardness were also performed.

– During the curing, the rubber-containing phase precipitated from the solution, forming micro-sized particles. Volume fractions of the particles were shown to significantly exceed (3–10 times) the volume of the added rubber – a phenomenon not investigated in depth before.

– This posits that the rubber-containing phase is not a rubber-only or even a rubber-rich phase. These findings are further substantiated by the results of DSC and microhardness tests – a phase or phases with glass-transition temperatures and hardness intermediate between rubber and polyester resin or polystyrene are present. Multiple phases with different rubber richness would better fit the experimental results.

– Further evidence for multiple phases is found in the internal differentiation of precipitated particles. Particles in both VTBNX-modified compositions (as evidenced by SEM) and high rubber content ETBN-modified composition (as evidenced by visual microscopy) are shown to be inhomogeneous. A model of the internal structure of the particles was proposed.

– The additional substance in the rubber-containing phase may be inferred to be polymerization or co-polymerization product of the styrene coming from the UP resin. In compositions with high rubber content, this may lead to a styrene-poor polyester matrix. Since styrene in UP resins performs an essential function in the curing reaction, a styrene deficit may lead to incomplete curing. This may or may not cause deterioration of the matrix properties.

– The size of the precipitated particles is dependent on the type of rubber used and the rubber content. ETBN forms significantly smaller particles than VTBNX. Smaller-sized particles should be, according to previous finds of various researchers, beneficial for the toughness of the compositions.

– Particles in both VTBNX- and ETBN-modified compositions show good adhesion to the polyester matrix – in the case of VTBNX, this is contrary to the findings by some other researchers, who used the same rubber with vinyl ester resin.

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- nowe surowce poliolowe i izocyjanianowe
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- katalizatory i inne substancje pomocnicze
- nowoczesne rozwiązania technologiczne
- poliuretany wytwarzane metodami bezizocyjanianowymi
- modyfikacja kompozycji PUR
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