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Characterization of self-adhesive structural tapes modified with polyvinyl acetal resins

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ABSTRACT

Thermally curable self-adhesive structural tapes (SATs) based on epoxy resin/acrylate copolymer composition and modified with three types of polyvinyl acetal resins (i.e. polyvinyl butyral-*stat*-acetal and polyvinyl butyrals) were prepared. The influence of polyvinyl resins addition on reactivity and self-adhesive features of SATs as well as on mechanical resistance of aluminum-SAT-aluminum joints was investigated. It was revealed that polyacetal resin addition (0.5 wt. part/100 wt. parts of adhesive composition) increases the adhesion of SATs to a steel substrate and, moreover, improves the overlap shear strength of thermally cured joints. The type and content of applied polyvinyl acetals influence the enthalpy of the photocrosslinking process, crosslinking degree as well as the epoxy groups conversion of SATs matrix.

1. Introduction

Self-adhesive structural tapes (SATs) are a new type of heavy duty structural adhesives in form of pressure-sensitive adhesive [1]. First SATs were developed in 1998 by 3M Company as structural bonding tapes (SBT) [2–4]. These materials were based on thick self-adhesive opaque films (> 0.5 mm) and exhibited relatively high shear strength after thermal curing. SBT tapes were designed to bond metallic elements in automotive and aircraft industries. Highly transparent epoxy-based thermosetting SATs have been developed by the authors team [1] and were characterized by thickness of 25–100 μm, good adhesion to steel, aluminum and glass, long pot-life at room temperature as well as high shear strength (after curing). Taking into consideration that relatively cheap polyvinyl acetal resins (polyacetals) exhibit high tensile strength, elongation at break, thermal resistance [5] and effectively improve adhesion of organic coatings and liquid adhesive to various substrates [6–8], these polymeric materials were tested as components of the SATs. Generally, hydroxyl functional polyacetals are produced by condensation of polyvinyl alcohol with n-butyraldehyde (polyvinyl butyral, PVB) and/or acetaldehyde (e.g. polyvinyl butyral-*stat*-acetal, PVBA) in the presence of an acid catalyst [6,9]. The mentioned hydrophobic polymers are also utilized as crosslinkable binders [6] and surface shrinkage modifiers for organic compositions [10], but the main application of polyacetals is safety laminated glass production, particularly for automotive, aerospace and architectural purposes [6,11]. This paper presents influence of selected polyvinyl acetal resins (PVB, PVBA) on reactivity (epoxy group conversion, crosslinking degree), self-adhesive (adhesion to steel, cohesion) and mechanical (shear strength) properties of thermally curable pressure-sensitive structural adhesives.

2. Experimental

2.1. Materials

The following components were used for preparation of an acrylate copolymer for the self-adhesive structural tapes (SATs): n-butyl acrylate (BA), glycidyl methacrylate (GMA), 2-hydroxyethyl acrylate (HEA) (BASF, Germany), 4-acryloyloxy benzophenone (ABP; Chemitec, Germany), 2,2′-azobis(isobutyronitrile) (AIBN; Merck, Germany) and ethyl acetate (EA; POCh, Poland) as a solvent. Bisphenol-A type liquid epoxy resin with epoxy equivalent weight ca. 188 g/equiv. and viscosity 12,500 mPa s (Organika-Sarzyna, Poland) was used as an epoxy component of the SATs. Additionally, the Lewis acid adduct (Nacure Super Catalyst A 218; Worleé-Chemie, Germany) and modified polymethylalkylsiloxane (BYK 325; BYK-Chemie, Germany) were applied as latent curing agent and adhesion promoter, respectively. Polyvinyl butyral-*stat*-

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Table 1
Specification of polyvinyl acetals [12].

Name	Symbol	Type	PVOH[c] block content (wt%)	PVAc[d] block content (wt%)	Tg[e] (°C)	Average molecular weight (g/mole)[f]	Viscosity (mPa s)[g]
Pioloform BL16	P-BL16	PVBA[a]	16.0	2.5	84	20,000	19
Mowital B20 H	M-B20	PVB[b]	19.5	2.5	64	20,000	24
Mowital B30 HH	M-B30	PVB	12.0	2.5	63	30,000	29

[a] Polyvinyl butyral-*stat*-acetal;
[b] Polyvinyl butyral;
[c] Polyvinyl alcohol;
[d] Poly(vinyl acetate);
[e] Glass transition temperature (DSC technique, ISO 11357-1);
[f] Weight average molecular weight;
[g] For 10 wt% solution of a polyvinyl resin in ethanol/toluene equiweight mixture (20 °C, Brookfield method, 30 rpm).

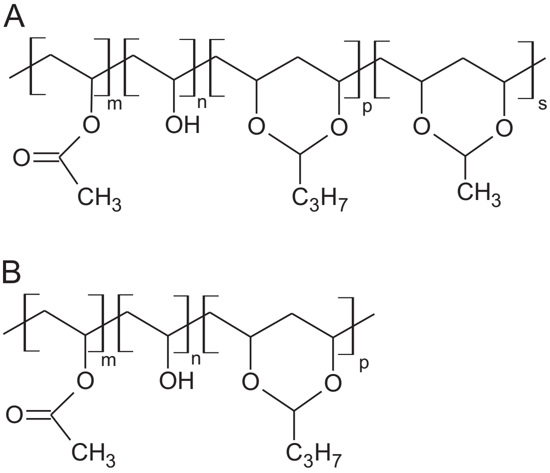

Fig. 1. Chemical structure of tested PVBA (A) and PVB (B).

acetal Pioloform BL16 (P-BL16) as well as polyvinyl butyrals Mowital B20 H (M-B20) and Mowital B30 HH (M-B30) (Kuraray Europe, Germany) were used as polymeric modifiers of the SAT composition. Characteristics and chemical structures of the applied polyvinyl resins are presented in Table 1 and Fig. 1, respectively.

2.2. Preparation and characterization of the acrylate copolymer

The acrylate copolymer was synthesized via free radical copolymerization of BA (61 wt%), GMA (25 wt%), HEA (14 wt%) and ABP (1.0 wt%) in EA containing AIBN (0.1 wt. part/100 wt. parts of monomers). The copolymerization process was carried out for 5 h at 78 °C in a glass reactor equipped with a mechanical stirrer and oil bath. Monomers and initiator were applied without their purification.

2.3. Preparation of SATs

The structural adhesives tapes were compounded using the acrylate copolymer (50 wt. parts), epoxy resin (50 wt. parts), latent curing agent (1 wt. part) as well as silicone adhesion promoter (0.5 wt. part) and polyvinyl acetal resin (0.1–5.0 wt. parts). The adhesive compositions were applied onto a silicone paper or polyester film (samples for self-adhesive tests), dried for 10 min at 105 °C and then UV-C irradiated using the medium pressure mercury lamp (Hönle UV-Technology, Germany). The UV exposition was controlled with the integrated radiometer (Dynachem 500, Dynachem Corp., USA); a single UV dose was 50 mJ/cm^2. Base weight and thickness of the UV-photocrosslinked adhesive layers were 120 g/m^2 and 100 µm, respectively. Description of the prepared structural adhesive tapes is shown in Table 2.

2.4. Self-adhesive tests of SATs

Self-adhesive properties of the prepared SATs were tested according to AFERA standards (Association des Fabricants Européens de Rubans Auto-Adhésifs), i.e. AFERA 4001 (adhesion to a steel substrate) and AFERA 4012 (cohesion). These parameters were evaluated using three samples of each SAT. Adhesion is defined as a force value required to remove a pressure sensitive material from stainless steel plate; the removal is proceed at the angle of 180° with speed of 300 mm/min [13]. Cohesion (static shear adhesion) describes the time needed to shear off the adhesive tape sample (under load of 1 kg) from defined steel surface.

2.5. Thermal analysis of SATs and characterization of prepared joints

Differential scanning calorimeter (DSC Q100, TA Instruments, USA) was used for determination of glass transition temperature (T_g) of SATs as well as enthalpy of their curing process (ΔH), onset temperature of curing reaction (T_i) and maximum/peak temperature of curing reaction (T_p). Standard aluminum DSC pans were used and samples (ca. 10 mg) were analyzed from −90 °C to 300 °C (with heating rate of 10 °C/min). Two DSC measurements for each composition were carried out. The overlap shear strength of aluminum-SAT-aluminum joints, prepared using degreased 2024 aluminum panels ($100 \times 25 \times 2$ mm^3) and cured at 160 °C for 40 min, was measured according to PN-ISO 4587 standard (ten samples of each system) at room temperature by means of Zwick/Roell Z010 machine (Zwick, Germany). Thermally cured SATs were also analyzed with the Fourier transform infrared spectroscope equipped with attenuated total reflectance (ATR) accessories (Nexus FT-IR, Thermo Nicolet, USA); variation of absorbance values at 915 cm^{-1} (oxirane groups) had been recorded and epoxy groups conversion (EGC) was calculated according to the equation [14]:

$$\mathrm{EGC} = \left(1 - \frac{A(t)}{A(0)}\right) \cdot 100\ (\%) \tag{1}$$

where: $A(0)$ – the initial intensity of the peak at 915 cm^{-1}; $A(t)$ – the intensity of the peak at 915 cm $^{-1}$ after thermal curing. The mentioned peaks were normalized in relation to the reference peaks at 1182 cm^{-1} (C–O–Ar bonds of bisphenol glicydyl ethers [15]).

Additionally, the crosslinking degree (α) of thermally cured SATs was calculated using DSC data according to the following equation [16]:

$$\alpha = \left(\frac{\Delta H_T - \Delta H_{res}}{\Delta H_T}\right) \tag{2}$$

where: ΔH_T – total enthalpy of SAT curing process (J/g); ΔH_{res} – enthalpy of post-curing process of the thermally cured SAT (in Al–SAT–Al joint).

Table 2
Composition of structural self-adhesives tapes with polyvinyl acetals.

SAT symbol	Components of SAT (wt. parts)					
	Polyvinyl acetal		Acrylate copolymer	Epoxy resin	Curing agent	Adhesion promoter
	Symbol	Dose[a]				
SAT-0	–	0	50	50	1	0.5
SAT-BL/01	P-BL16	0.1				
SAT-BL/025		0.25				
SAT-BL/05		0.5				
SAT-BL/1		1.0				
SAT-BL/25		2.5				
SAT-BL/50		5.0				
SAT-B20/01	M-B20	0.1	50	50	1	0.5
SAT-B20/025		0.25				
SAT-B20/05		0.5				
SAT-B20/10		1.0				
SAT-B20/25		2.5				
SAT-B20/50		5.0				
SAT-B30/01	M-B30	0.1	50	50	1	0.5
SAT-B30/025		0.25				
SAT-B30/05		0.5				
SAT-B30/10		1.0				
SAT-B30/25		2.5				
SAT-B30/50		5.0				

[a] wt. parts.

Microscopic images of thermally cured SATs (after mechanical test of Al–SAT–Al joints) were created using 3D Laser Scanning Microscope (LSM) VK-9700 (Keyence, USA).

3. Results and discussion

In this study two basic self-adhesive features (cohesion and adhesion) were investigated. Although cohesion values for the all UV-photocrosslinked SATs were similar and exceeded the threshold required for pressure-sensitive adhesives (i.e. 72 h at RT), values of adhesion to a steel substrate were diversified. As it can be seen in Fig. 2, the latter feature depended on a kind and content of polyvinyl resin in a SAT composition.

Generally, addition of 0.1–1.0 wt. part of a polyvinyl acetal significantly increased adhesion of SATs and the highest values of that parameter were noted for the samples with 0.5 wt. part of polyacetal. It is noteworthy that ca. 60% of adhesion increment was registered for the SATs with P-BL16 (16.4 N/25 mm, SAT-BL/05) or M-B30 (16.2 N/25 mm, SAT-B30/05) in relation to the reference sample (10.1 N/25 mm, SAT-0); SAT with M-B20 reached slightly lower adhesion (15.4 N/25 mm, SAT-B20/05). Probably, that phenomenon was caused by relatively higher acetal blocks content in P-BL16 (81.5 wt%, Table 1) and M-B30 (85.5 wt%) than in M-B20 (78 wt%). Moreover, the polyvinyl acetals were compounded with a liquid epoxy resin used for SATs preparation and that component might act as a plasticizer of PVA and PVBA [17]. It is known that chemical structure (mainly acetalization degree) of a polyvinyl resin and a type of plasticizer (i.e. its compatibility with the resin) affect its adhesion to various substrates [18–22]. Nevertheless, it should be noted that a higher dose of P-BL16 (> 1 wt%) markedly decreased adhesion of the prepared SATs. In this case reduction of adhesion could be explained by a higher T_g value of the mentioned additive (84 °C, Table 1) in relation to M-B20 (64 °C) and M-B30 (63 °C). It is known that T_g of a polymeric tape affects its pressure-sensitive features. Unfortunately, recorded DSC data does not confirm the presented thesis. Generally, the glass transition temperature values were similar for the all UV-photocrosslinked SATs containing polyvinyl resins (Table 3) and, moreover, the lowest values of that parameter (ca. 2–3 °C decline in respect to SAT-0) were observed for samples with the lowest (0.1 and 0.25 wt. part) and the highest (2.5 and 5 wt. parts) doses of the modifier. Interestingly, SATs containing 0.5 or 1 wt. part of a polyacetal (i.e. SATs with the highest adhesion to a steel substrate; Fig. 2) exhibited similar T_g value to the reference sample (– 12 °C).

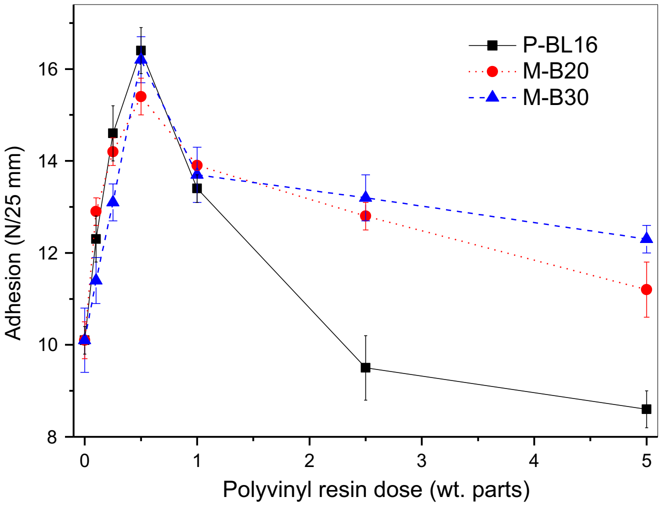

Fig. 2. Adhesion of SATs with polyvinyl acetals.

Although onset temperature of thermal curing reaction (T_i) and maximum temperature of the exothermic peak (T_p) were slightly affected by polyvinyl resin addition (Table 3), enthalpy of the curing process (ΔH) was markedly higher (in relation to SAT-0) for SATs with a polyvinyl resin. Moreover, the highest values of ΔH were registered for tapes with low content of the polymeric modifier. It shows that polyvinyl acetals addition (especially a small dose) influences on thermal curing process of UV-photocrosslinked epoxy-acrylate pressure sensitive tapes.

Investigated polyvinyl resins markedly affected overlap shear strength of thermally cured aluminum-SAT-aluminum joints (Fig. 3.). It should be noted that the highest values of that parameter were observed for the tapes characterized by the highest adhesion to a steel substrate before thermal curing process (i.e.

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Table 3
Thermal properties of UV-photocrosslinked SATs with polyvinyl acetals.

SAT symbol	Enthalpy, (ΔH) (J/g)	Onset reaction temperature (T_i) (°C)	Max. exothermic peak temperature (T_p) (°C)	Glass transition temperature (T_g) (°C)
SAT-0	224 ± 3	115 ± 1	173 ± 2	−12 ± 2
SAT-BL/01	278 ± 2	114 ± 2	178 ± 1	−14 ± 1
SAT-BL/025	273 ± 3	112 ± 2	176 ± 3	−15 ± 3
SAT-BL/05	238 ± 4	113 ± 1	180 ± 2	−11 ± 2
SAT-BL/10	264 ± 6	113 ± 2	177 ± 3	−13 ± 2
SAT-BL/25	243 ± 5	114 ± 1	178 ± 5	−15 ± 3
SAT-BL/50	220 ± 5	115 ± 1	180 ± 2	−14 ± 2
SAT-B20/01	274 ± 3	112 ± 2	176 ± 4	−15 ± 1
SAT-B20/025	259 ± 5	112 ± 3	180 ± 2	−15 ± 3
SAT-B20/05	251 ± 6	114 ± 1	177 ± 3	−12 ± 3
SAT-B20/10	261 ± 4	114 ± 2	178 ± 2	−12 ± 2
SAT-B20/25	251 ± 3	114 ± 3	177 ± 3	−13 ± 1
SAT-B20/50	247 ± 3	113 ± 2	177 ± 2	−14 ± 2
SAT-B30/01	255 ± 4	114 ± 2	175 ± 1	−14 ± 3
SAT-B30/025	290 ± 3	109 ± 3	189 ± 4	−16 ± 2
SAT-B30/05	260 ± 2	112 ± 2	174 ± 3	−13 ± 3
SAT-B30/10	240 ± 5	116 ± 1	178 ± 3	−13 ± 3
SAT-B30/25	226 ± 4	116 ± 4	177 ± 5	−14 ± 1
SAT-B30/50	257 ± 6	115 ± 2	177 ± 2	−15 ± 3

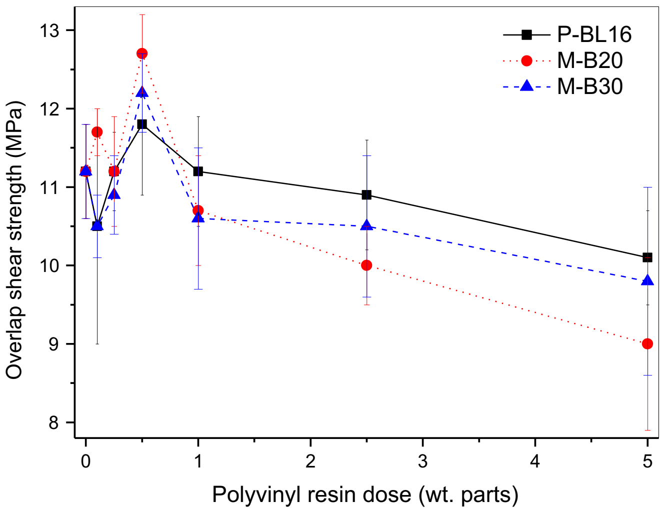

Fig. 3. Influence of polyvinyl acetal content on overlap shear strength of aluminum-SAT-aluminum joints.

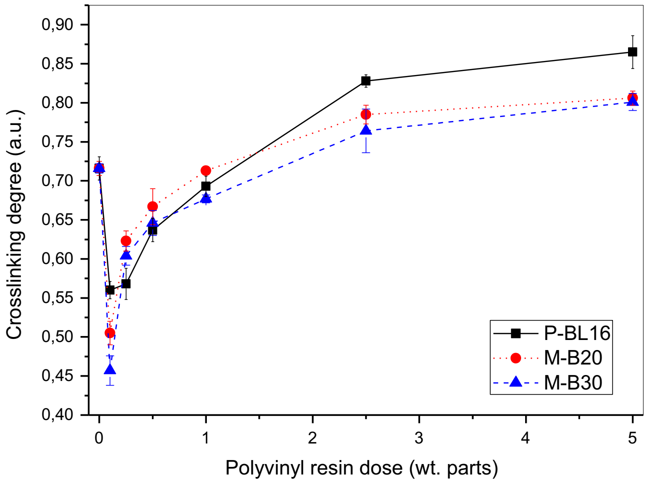

Fig. 4. Crosslinking degree for SATs with polyvinyl acetals (after a thermal curing process).

SATs with 0.5 wt. part of polyacetal; Fig. 3). Nevertheless, the samples with PVB (SAT-B20/05, SAT-B30/05) reached higher shear strength (12.7 MPa and 12.2 MPa, respectively) than SAT containing PVBA resin (SAT-BL/05, 11.8 MPa). In the case of higher doses of polyvinyl acetal resin (>2.5 wt. parts), samples with PVBA exhibited relatively higher mechanical resistance than samples with PVB resins.

Interestingly, overlap shear strength values of thermally cured aluminum-SAT-aluminum joints correlate with crosslinking degree of SATs (Fig. 4). Modified samples exhibiting extraordinary lower or higher crosslinking degree (in relation to the reference sample; 0.72 unit) reached generally lower shear strength than SAT-0 (Fig. 3). Considering SAT with the highest mechanical resistance (0.5 wt. part of M-B20; SAT-B20/05), it should be noted that the mentioned sample was characterized by a slightly higher crosslinking degree (0.67 unit) as well as higher epoxy group conversion (EGC) (27%; Fig. 5) in relation to the other tapes with the same content of polyacetals (0.64 unit and 26% for SAT-BL/05, 0.65 unit and 24% for SAT-B30/05). On the other hand, EGC values do not correlate with a crosslinking degree of thermally cured SATs (the similar conclusions were presented in the literature [10,23]).

As can be seen in Fig. 5, conversion of epoxy groups markedly increased with increasing polyacetal content in SATs; in the case of SAT-B20/50 ca. 80% of increment (in relation to SAT-0) was recorded. As mentioned before, M-B20 resin contains a much higher amount of hydroxyl groups (19.5 wt% of polyvinyl alcohol blocks content; Table 1) than the other modifiers (16 wt% for P-BL16, 12 wt% for M-B30). It is generally known that OH groups actively participate in a cationic polymerization process of epoxy resins and affect EGC value [10,23]. Epoxy/hydroxyl groups molar ratio may also influence on hardness, flexibility as well as on adhesion strength and tensile strength of the cured materials [23–26]. Nevertheless, too high concentration of a hydroxyl functional component in an epoxy composition (exceeding an optimal value) may significantly diminish its mechanical features [24,26]. In the case of investigated systems, it seems that the optimal content of polyvinyl resin (mainly M-B20) is 0.5 wt. part/100 wt. parts of SAT composition.

Considering LSM images of the reference sample (SAT-0) and the sample with the highest M-B20 resin content (5 wt. parts; SAT-B20/50) (after their mechanical test; Fig. 6), it could be claimed that the latter material is a quite uniform, i.e. PVB is completely dissolved in the SAT matrix. Moreover, a ductile fracture of SAT-B20/50 was observed (and a brittle fracture for SAT-0). It means

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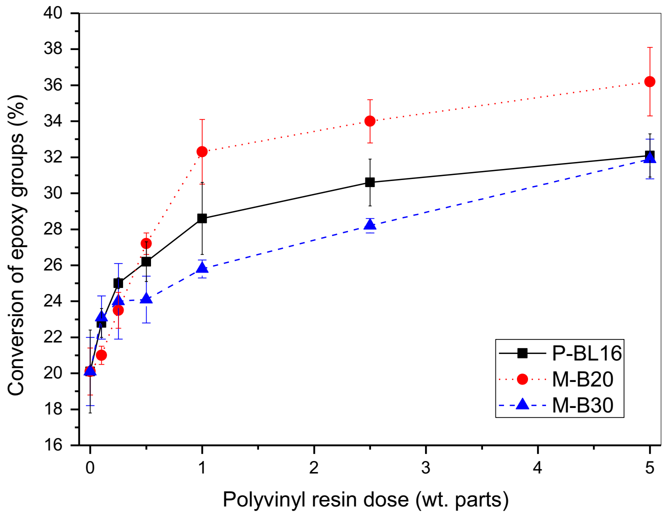

Fig. 5. Epoxy groups conversion for SATs with polyvinyl acetals (after a thermal curing process).

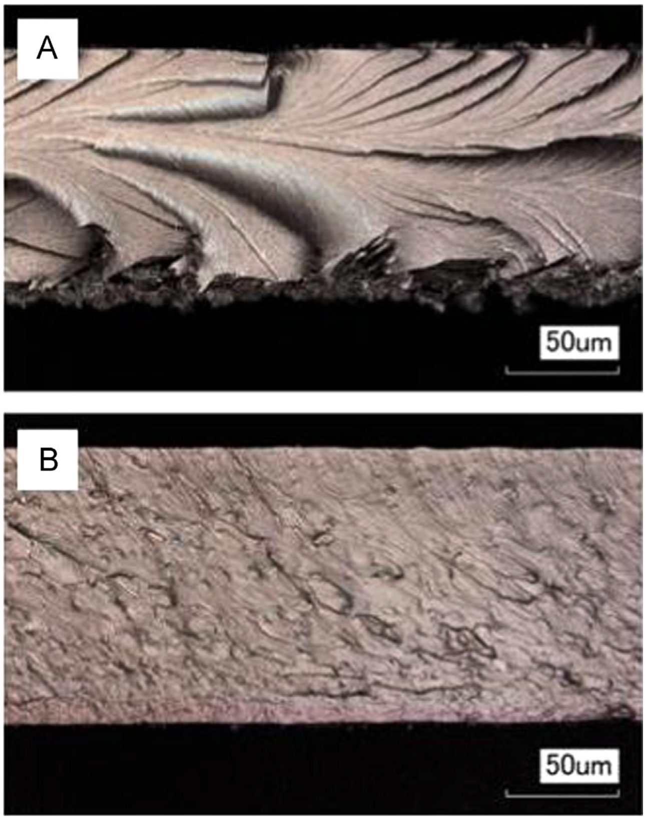

Fig. 6. LSM images of cured SAT joints: reference sample (SAT-0) (A) and sample with 5 wt. parts of Mowital B20 H (SAT-B20/50) (B).

that polyvinyl resin act as a effective plasticizer of thermally cured epoxy-acrylate composition [17] used for SATs preparation.

4. Conclusion

In this paper, influence of polyvinyl acetals (i.e. polyvinyl butyral-*stat*-acetal or polyvinyl butyrals) addition on self-adhesive and mechanical properties of epoxy-acrylate structural adhesive tape (SAT) has been presented and analyzed. Based on the test results the following conclusions can be drawn:

– Polyacetals addition (< 1 wt. part/100 wt. parts of adhesive composition) significantly improves adhesion of the SAT to a steel substrate. Probably, that feature is mainly affected by acetalization degree and/ or compatibility of the polyvinyl acetals with epoxy-acrylate component of the SAT.

– A dose of a polyvinyl acetal significantly impacts crosslinking degree and epoxy groups conversion of thermally cured SAT. Moreover, the latter parameter directly depends on polyvinyl alcohol blocks content in the polymeric modifier.

– SATs containing 0.5 wt. part of the polyvinyl acetal resins reached higher mechanical feature (i.e. overlap shear strength of aluminum-SAT-aluminum joints; 11.8–12.7 MPa) in comparison with the unmodified SAT (11.2 MPa).

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