



## EFFECT OF METAL CATALYSIS AND FUNCTIONALITY OF A METALLOSILOXANE DURING THE FORMATION OF METALLOSILOXANE–EPOXY MATERIALS

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### Abstract

The catalytic activity of a wide range of metals incorporated into the structures of metallosiloxane oligomers in the curing process of epoxy-diane resins has been estimated. From the viewpoint of activity, the most preferable metallosiloxanes are those containing Al, Hf, and Nb, and from the viewpoint of cost—Al, Ti, Fe<sup>II</sup>, Zr. It is shown that the structure and functionality of the metallosiloxane can serve as the factors to control the formation of a metallosiloxane–epoxy material, which allows for increasing the efficiency of metal catalysis.

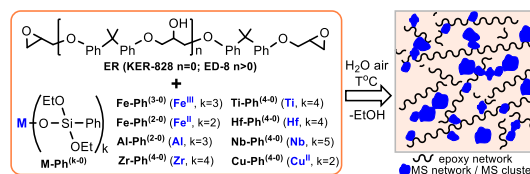
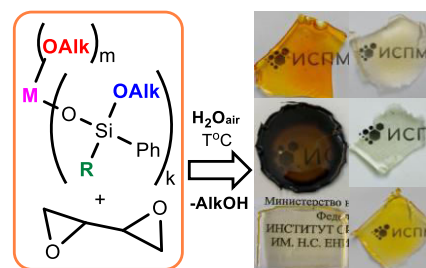
**Key words:** metallosiloxanes, epoxy resins, curing, metal catalysis, structure–properties relationships.

### Introduction

Epoxy resins are among the most popular matrices for the creation of polymer composite materials, with epoxy-diane resins being the most widely used ones, and the optimization of their curing and filling methods is still an important task [1]. It is known that the introduction of siloxane and/or metal oxide components into an epoxy material improves its thermomechanical characteristics [2, 3]. Earlier we have demonstrated the efficiency of using partially diethoxy(phenyl)siloxy-substituted metallosiloxane oligomers (MSs) as the curing agents and fillers for epoxy-diane resins (ERs) [4]. This approach enabled the production of the compositions that were homogeneous at the micro level in a wide range of the filling degrees with the MS component. The nature of the metal atom and the MS structure can affect the efficiency of the curing process. In this work, the catalytic activity of a number of metals and the effect of the structure and composition of the curing agent on the curing of epoxy-diane resins were studied using the example of the alkoxy(phenyl)siloxy derivatives of MS oligomers.

### Results and discussion

The catalytic activity of the metal atom in the MS structure was studied in the opening of epoxy groups of fully diethoxy(phenyl)siloxy-substituted derivatives **M-Ph**<sup>(k-0)</sup>. The resins in use, KER-828 and ED-8, differed in molecular weight and, consequently, in the number of epoxy groups, and the presence of a certain initial content of hydroxy groups in the system (Fig. 1).



**Figure 1.** Structures of the MS oligomers and epoxy resins in use and the scheme of the formation of the MS–epoxy materials.

The formation of the materials was carried out according to the earlier described method with the heating mode of 70 °C (1 h) → 100 °C (1 h) → 150 °C (2 h), which allowed us to compare the newly obtained data with the previous ones [4]. The gel fraction (GF) value and a decrease in the intensity of an absorption band at 915 cm<sup>-1</sup> in the IR spectra, which corresponds to the closed epoxy ring, were used as a measure of the curing efficiency. The qualitative features were homogeneity (transparency) and strength (fracture) of the samples obtained (Table 1).

With respect to the [M]:[epoxy] ratios, the following series of catalytic activity of the metals explored in the epoxy ring opening can be outlined: Al >> Hf > Nb > Ti ≥ Fe<sup>II</sup> > Zr > Fe<sup>III</sup> > Cu<sup>II</sup>. This is demonstrated graphically in Fig. 2 that highlights the region corresponding to the minimum MS consumption for achieving GF ≥ 98 wt %, which includes MSs containing Al, Hf, and Nb.

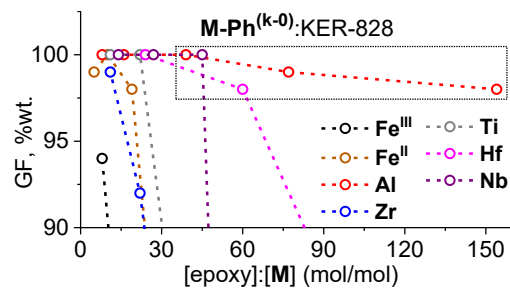
In general, the use of larger initial amounts of MSs is not unreasonable, since this significantly increases the fracture strength of the composite. In some cases, however, this can lead to heterogeneity (Zr- and Nb-siloxanes), as well as when using ED-8 resin (Fig. 3).

**Table 1.** GF values of the samples obtained upon curing of the initial system  $\{M\text{-Ph}^{(k-0)}\text{:ER} = x\text{:}1 \text{ wt. pt.}\}$  and the calculated molar ratios of the metal and ethoxy groups to the epoxy groups in the initial cured system

M	ER	x, wt. pt.	[M]:[epoxy]	[OEt]:[epoxy]	GF, wt %	H	S		
$\text{Fe}^{\text{III}}$	KER-828	0.05	1:82	1:14	0	+	-		
		0.10	1:41	1:7	53	+	-		
		0.25	1:16	1:3	80	+	-		
		0.50	1:8	1:1	94	+/-	+		
		ED-8	0.25	1:6	1:1	98	+	+	
$\text{Fe}^{\text{II}}$	KER-828	0.10	1:28	1:7	83	+	+		
		0.15	1:19	1:5	98	+	++		
		0.25	1:10	1:3	>99	+	++		
		0.50	1:5	1:1	99	+/-	++		
		ED-8	0.10	1:11	1:3	94	+	+	
	ED-8	0.15	1:8	1:2	>99	+	+		
		0.25	1:4	1:1	>99	+	+		
		0.50	1:2	1:0.4	>99	+/-	-		
		$\text{Al}$	KER-828	0.025	1:154	1:26	98	+	+
				0.05	1:77	1:13	99	+	+
0.10	1:39			1:7	>99	+	+		
0.25	1:16			1:3	>99	+	++		
0.50	1:8			1:1	>99	+	++		
	ED-8	0.05	1:31	1:5	97	-	+		
		$\text{Zr}$	KER-828	0.05	1:111	1:14	44	+	-
				0.10	1:56	1:7	50	+	-
				0.25	1:22	1:3	92	+	+
				0.50	1:11	1:1	99	-	++
ED-8	0.10			1:22	1:3	85	-	-	
$\text{Ti}$	KER-828	0.05	1:108	1:13	51	+	-		
		0.10	1:54	1:7	72	+	-		
		0.15	1:36	1:4	83	+	+		
		0.25	1:22	1:3	>99	+	-		
		0.50	1:11	1:1	>99	+	+		
	ED-8	0.05	1:43	1:5	62	+	-		
		0.10	1:22	1:3	89	+	+		
		0.25	1:9	1:1	95	+	-		
		$\text{Hf}$	KER-828	0.05	1:120	1:15	77	+	-
				0.10	1:60	1:8	98	+	+
0.25	1:24			1:3	>99	+	++		
ED-8	0.05			1:48	1:6	>99	-	-	
0.10	1:24			1:3	>99	+	++		
$\text{Nb}$	KER-828	0.10	1:68	1:7	0	+	-		
		0.15	1:45	1:5	>99	+	++		
		0.25	1:27	1:3	>99	+	+		
		0.50	1:14	1:1	>99	-	+		
		ED-8	0.05	1:54	1:6	93	+	+	
	ED-8	0.10	1:27	1:3	>99	+	++		
		0.25	1:11	1:1	>99	-	+		
		$\text{Cu}^{\text{II}}$	KER-828	0.50	1:6	1:2	9	+	-
1	1:3			1:1	13	+	-		
ED-8	0.50			1:2	1:1	49	-	-	

H is the composite homogeneity; S is the composite strength.

The use of ED-8 generally allows for reducing the MS consumption to achieve a high degree of cross-linking, which is explained not only by the reduced content of epoxy groups in the initial system, but also by the more intense cross-linking of the MS and epoxy networks by their functional groups. Both facts are illustrated by the IR spectra (Fig. 4). Thus, for system  $\{\text{Hf-Ph}^{(4-0)}\text{:KER-828} = 0.1\text{:}1 \text{ wt. pt.}\}$ , a noticeable residual



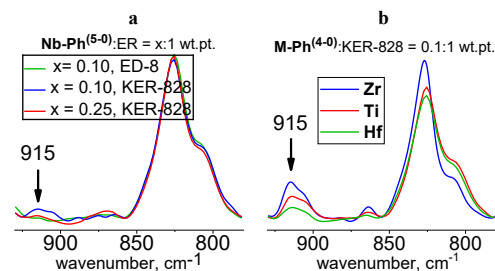
**Figure 2.** Graphic illustration of the efficiency of metal catalysis for  $M\text{-Ph}^{(k-0)}\text{:KER-828}$  systems.



**Figure 3.** Examples of  $\{MS:ER = x\text{:}1 \text{ wt. pt.}\}$  compositions.

intensity of the absorption band at  $915 \text{ cm}^{-1}$  was observed even at  $GF = 98 \text{ wt. \%}$ , which indicates the occurrence of curing also due to the interaction of the MS and epoxy networks. This can also explain the lower GF values in the case of fully siloxy-substituted  $\text{Fe-Ph}^{(3-0)}$  and  $\text{Zr-Ph}^{(4-0)}$  compared to the previously obtained data for their partially siloxy-substituted analogs [4]. The step-like nature of the GF dependence on the MS content observed for system  $\{\text{Nb-Ph}^{(5-0)}\text{:KER-828}\}$  also suggests a significant role of such a process in this case. The same can be noted for system  $\{\text{Cu-Ph}^{(2-0)}\text{:ER}\}$ , where copper almost does not catalyze the opening of epoxy rings, and in the case of ED-8 it facilitates only partial binding of the networks. Thus, metal catalysis is also important for this process. In general, the intensity of the absorption band at  $915 \text{ cm}^{-1}$  strongly correlates with the GF values.

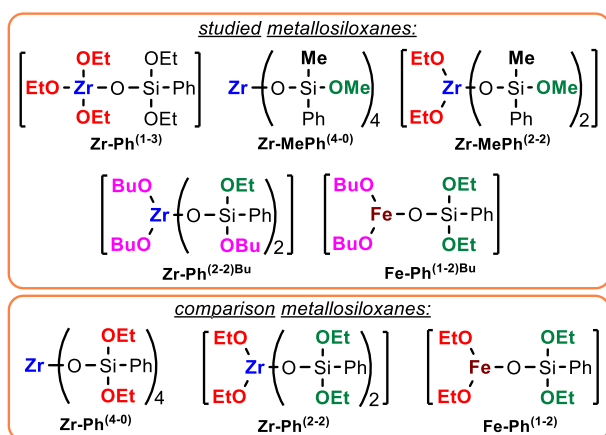
Therefore, the MS ability to bind with the epoxy matrix and, therefore, its functionality and activity in the formation of its own cross-linked network play important roles in the curing efficiency.



**Figure 4.** Selected IR spectra of the initial composition  $\{MS:ER = x\text{:}1 \text{ wt. pt.}\}$ .

For a more detailed study of these factors, a number of phenyl-containing MSs were additionally synthesized: **Zr-Ph**<sup>(1-3)</sup>, **Zr-MePh**<sup>(4-0)</sup>, **Zr-MePh**<sup>(2-2)</sup>, **Fe-Ph**<sup>(1-2)Bu</sup>, and **Zr-Ph**<sup>(2-2)Bu</sup>, differing in the presence, quantity and type of alkoxy groups from the previously synthesized analogs **Zr-Ph**<sup>(2-2)</sup>, **Zr-Ph**<sup>(4-0)</sup>, and **Fe-Ph**<sup>(1-2)</sup> (Fig. 5).

The synthesis of these MSs was carried out similarly to the previously obtained analogs using MePh-SiONa and sodium butoxide. In this case, the yield >100% and the elemental analysis data for **Zr-Ph**<sup>(2-2)Bu</sup> indicate the occurrence of partial transalkoxylation of Si-OEt groups for Si-OBu in the presence of butanol in the reaction mixture, which was taken into account further in the calculations.



**Figure 5.** Structures of the zirconium and iron siloxanes and comparison objects studied as the ER curing agents.

They were used as the curing agents for KER-828 resin, and the GF data were compared with those for their analogs (Table 2). By comparing the GF values and the calculated molar ratios [M]:[epoxy] and [OR]:[epoxy], it is possible to construct the following series of zirconium and iron siloxanes in terms of their catalytic activity: **Zr-Ph**<sup>(2-2)Bu</sup> > **Zr-Ph**<sup>(2-2)</sup> > **Zr-Ph**<sup>(4-0)</sup> > **Zr-Ph**<sup>(1-3)</sup> ≥ **Zr-MePh**<sup>(2-2)</sup> > **Zr-MePh**<sup>(4-0)</sup> and **Fe-Ph**<sup>(1-2)Bu</sup> > **Fe-Ph**<sup>(1-2)</sup> > **Fe-Ph**<sup>(3-0)</sup>. The lower activity in the case of **Zr-Ph**<sup>(1-3)</sup> can be associated with the tendency of the MS oligomer to more rapidly form its own cross-linked network and thereby shield the ligand sphere of the metal atom due to a higher proportion of hydrolytically active Zr-OEt groups, and in the case of **Zr-MePh**<sup>(4-0)</sup> and **Zr-MePh**<sup>(2-2)</sup>—with reduced alkoxy functionality and, possibly, with the greater shielding of the metal atom. The most effective systems were butoxy derivatives **Fe-Ph**<sup>(1-2)Bu</sup> and **Zr-Ph**<sup>(2-2)Bu</sup>. This fact cannot be associated with the greater shielding of the metal atom by bulkier butoxy groups. In this case, the factor of the rate of formation of its own network is clearly evident: here it should be lower than for the ethoxy analogs due to a decrease in the hydrolytic activity of metal butoxy groups. This means that the MS lifetime in the oligomeric form is increased. In addition, this also affects the activity in binding to the polymerizing epoxy matrix. Both factors are confirmed by a slight decrease in the GF value at the highest initial MS content in the case of using **Fe-Ph**<sup>(1-2)Bu</sup>, i.e., some fractions of the resulting MS network are soluble, as well as a decrease in the GF values when using ED-8 resin, which initially contains hydroxy groups capable of binding to the growing MS network (see Table 2). In general, an increase in

the content of alkoxy groups in the initial system also contributes to an increase in the curing degree. Figure 3 also shows the appearance of some of the resulting samples, reflecting the general trends in the compatibility of the material components.

**Table 2.** Comparative GF values of the samples obtained during curing of the initial system {MS:KER-828 = x:1 wt.pt.}, as well as the calculated molar ratios of the metal and alkoxy groups to epoxy groups in the initial cured system

MS	x, wt. pt.	GF, wt %	[M]:[epoxy]	[OR]:[epoxy]	H	S
<b>Zr-Ph</b> <sup>(4-0)</sup>	0.25	92	1:22	1:3	+	+
	0.50	99	1:11	1:1	–	++
<b>Zr-Ph</b> <sup>(2-2)</sup>	0.10	96 <sup>a</sup>	1:14 <sup>a</sup>	1:2 <sup>a</sup>	+ <sup>a</sup>	– <sup>a</sup>
	0.15	93	1:24	1:4	+	+
<b>Fe-Ph</b> <sup>(1-2)</sup>	0.25	99	1:14	1:2	+	+
	0.50	>99	1:4	1:1	+	++
<b>Zr-Ph</b> <sup>(1-3)</sup>	0.15	82	1:17	1:3	+	–
	0.25	99	1:10	1:2	+/-	+
<b>Zr-MePh</b> <sup>(4-0)</sup>	0.50	>99	1:5	1:1	–	++
	0.25	65	1:18	1:5	+	–
<b>Zr-MePh</b> <sup>(2-2)</sup>	0.50	82	1:9	1:2	+	–
	0.25	93	1:12	1:3	+	–
<b>Fe-Ph</b> <sup>(1-2)Bu</sup>	0.10	70 (79 <sup>a</sup> )	1:25 (1:10 <sup>a</sup> )	1:6 (1:2 <sup>a</sup> )	+ <sup>(a)</sup>	–(+ <sup>a</sup> )
	0.25	99 (89 <sup>a</sup> )	1:10 (1:4 <sup>a</sup> )	1:3 (1:1 <sup>a</sup> )	+	+(- <sup>a</sup> )
<b>Zr-Ph</b> <sup>(2-2)Bu</sup>	0.50	95 <sup>(a)</sup>	1:5 (1:2 <sup>a</sup> )	1:1 (1:0.4 <sup>a</sup> )	+/- (- <sup>a</sup> )	++ (- <sup>a</sup> )
	0.05	91 <sup>a</sup>	1:34 <sup>a</sup>	1:6 <sup>a</sup>	+ <sup>a</sup>	–*
<b>Zr-Ph</b> <sup>(2-2)Bu</sup>	0.10	92 (>99 <sup>a</sup> )	1:42 (1:17 <sup>a</sup> )	1:7 (1:3 <sup>a</sup> )	+	–(+ <sup>a</sup> )
	0.25	>99	1:17	1:3	+	+

H is the composite homogeneity; S is the composite strength;

<sup>a</sup> the values were obtained using ED-8 resin.

## Experimental section

### Methods and materials

The sample formation and GF determination were performed similarly to the previous studies [4]. The IR spectra were recorded on a Bruker Tensor 27 spectrometer (Germany).

Metal chlorides (purity >99.9%) were used without preliminary processing. Triethoxy(phenyl)silane and methyl(dimethoxy)phenylsilane (MDMPS) were distilled in an argon atmosphere prior to use. Epoxy-diane resins of KER-828 and ED-8 trademarks were used (the content of epoxy groups was 24.0–25.0 and 8.5–10.0 wt %, respectively). Toluene, ethanol, butanol, and ethyl acetate were dried by prolonged refluxing and distillation over CaH<sub>2</sub> under an argon atmosphere and stored over 3 Å molecular sieves. Ethyl acetate for measurements was distilled on a rotary evaporator.

### Syntheses

The syntheses of the following MSs were published in Refs. [5, 6]: tris-[diethoxy(phenyl)siloxy]aluminum (**Al-Ph**<sup>(3-0)</sup>), tris-[diethoxy(phenyl)siloxy]iron (**Fe-Ph**<sup>(3-0)</sup>), tetrakis-[diethoxy(phenyl)siloxy]zirconium (**Zr-Ph**<sup>(4-0)</sup>), tetrakis-[diethoxy(phenyl)siloxy]hafnium (**Hf-Ph**<sup>(4-0)</sup>), bis-[diethoxy(phenyl)siloxy]copper (**Cu-Ph**<sup>(2-0)</sup>), and triethoxy-[diethoxy(phenyl)siloxy]zirconium (**Zr-Ph**<sup>(1-3)</sup>).

Bis-[diethoxy(phenyl)siloxy]iron (**Fe-Ph**<sup>(2-0)</sup>), tetrakis-

[diethoxy(phenyl)siloxy]titanium (**Ti-Ph**<sup>(4-0)</sup>) and pentakis-[diethoxy(phenyl)siloxy]niobium (**Nb-Ph**<sup>(5-0)</sup>) were obtained similarly to the published procedures [5] by the interaction of FeCl<sub>2</sub>, TiCl<sub>4</sub>, and NbCl<sub>5</sub> with sodium diethoxy(phenyl)silanolate Ph-SiONa in toluene (in the case of **Ti-Ph**<sup>(4-0)</sup> and **Nb-Ph**<sup>(5-0)</sup>) and toluene/ethanol = 5/1 (v/v) (in the case of **Fe-Ph**<sup>(2-0)</sup>) at the equal molar contents [M-Cl]:[SiONa]. Dark brown and colorless viscous liquids were obtained, respectively, in 84, 82 and 81% yields. Anal. Calcd for **Fe-Ph**<sup>(2-0)</sup> C<sub>20</sub>H<sub>30</sub>O<sub>6</sub>Si<sub>2</sub>Fe: C, 50.21; H, 6.32; Si, 11.74; Fe, 11.67. Found: C, 45.35; H, 5.85; Si, 11.56; Fe, 11.52%. Anal. Calcd for **Nb-Ph**<sup>(5-0)</sup> C<sub>50</sub>H<sub>75</sub>O<sub>15</sub>Si<sub>5</sub>Nb: C, 52.25; H, 6.58; Si, 12.22; Nb, 8.08. Found: C, 46.29; H, 5.25; Si, 13.73; Nb, 9.63%.

**Sodium methyl(methoxy)phenylsilanolate (MePh-SiONa)** was obtained similarly to diethoxy(phenyl)silanolate by reacting sodium hydroxide with MDMPS in dry toluene at the ratio [NaOH]:[MDMPS] = 1:1 n/n until complete dissolution of sodium hydroxide at 50 °C. After removal of volatiles (1 Torr, 60 °C), the product was isolated as a white paste-like mass. The product yields varied within 97–99%.

**Tetrakis-[methyl(methoxy)phenylsiloxy]zirconium (Zr-MePh**<sup>(4-0)</sup>) was prepared according to the published procedure [5] by reacting a suspension of ZrCl<sub>4</sub> (2.07 g, 0.0089 mol) in 15 mL of dry toluene with a solution of MePh-SiONa (6.77 g, 0.0356 mol) in 30 mL of dry toluene. After separation of the precipitate, a clear colorless solution was obtained. The yield of the product was 5.72 g (84%). The dry substance was a transparent colorless highly viscous liquid. Anal. Calcd for C<sub>32</sub>H<sub>44</sub>O<sub>8</sub>Si<sub>4</sub>Zr: C, 50.56; H, 5.83; Si, 14.78; Zr, 12.00. Found: C, 50.91; H, 6.07; Si, 14.98; Zr, 11.79%.

**Diethoxy-bis-[methyl(methoxy)phenylsiloxy]-zirconium (Zr-MePh**<sup>(2-2)</sup>) was obtained according to the published procedure [7] by reacting a suspension of ZrCl<sub>4</sub> (3.03 g, 0.0130 mol) in 10 mL of dry toluene with a mixture of a solution of Na (0.60 g, 0.0260 mol) in 15 mL of dry ethanol and a solution of MePh-SiONa (5.20 g, 0.0260 mol) in 40 mL of dry toluene. After separation of the precipitate and distillation of the toluene–ethanol azeotropic mixture, a transparent colorless solution was obtained. The yield of the product was 5.98 g (89%). The dry substance was a colorless transparent highly viscous liquid. Anal. Calcd for C<sub>20</sub>H<sub>32</sub>O<sub>6</sub>Si<sub>2</sub>Zr: C, 46.57; H, 6.25; Si, 10.89; Zr, 17.68. Found: C, 42.47; H, 4.76; Si, 11.39; Zr, 18.95%.

**Di-*n*-butoxy[diethoxy(phenyl)siloxy]iron (Fe-Ph**<sup>(1-2)</sup>**Bu)** was obtained according to the published procedure [7] by reacting a suspension of FeCl<sub>3</sub> (6.42 g, 0.0395 mol) in 25 mL of dry toluene with a mixture of a solution of Na (1.82 g, 0.0790 mol) in a mixture of dry *n*-butanol (30 mL) and toluene (15 mL) and a solution of Ph-SiONa (9.50 g, 0.0395 mol) in 50 mL of dry toluene. After separation of the precipitate and distillation of the toluene–ethanol–butanol azeotropic mixture, a transparent dark brown solution was obtained. The yield of the product was 14.00 g (86%). The dry substance was a dark brown highly viscous liquid. Anal. Calcd for C<sub>18</sub>H<sub>33</sub>O<sub>5</sub>SiFe: C, 52.30; H, 8.05; Si, 6.79; Fe, 13.51. Found: C, 49.07; H, 7.00; Si, 5.97; Fe, 11.91%.

**Di-*n*-butoxy-bis-[diethoxy(phenyl)siloxy]zirconium (Zr-Ph**<sup>(2-2)</sup>**Bu)** was obtained according to the published procedure [7] by reacting a suspension of ZrCl<sub>4</sub> (5.80 g, 0.025 mol) in 25 mL of dry toluene with a mixture of a solution of Na (1.15 g, 0.050 mol) in a mixture of dry *n*-butanol (20 mL) and toluene

(10 mL) and a solution of Ph-SiONa (11.72 g, 0.050 mol) in 50 mL of dry toluene. After separation of the precipitate and distillation of the toluene–ethanol–butanol azeotropic mixture, a transparent solution was obtained. The product yield was 16.90 g (102%). The yield relative to the partially transalkoxylated product [OBu]<sub>2</sub>Zr[OSi(Ph)(OEt)(OBu)]<sub>2</sub> was 94%. The dry substance was a transparent orange viscous liquid. Anal. Calcd for C<sub>28</sub>H<sub>48</sub>O<sub>8</sub>Si<sub>2</sub>Zr: C, 50.95 (53.67); H, 7.33 (7.88); Si, 8.51 (7.84); Zr, 13.82 (12.74). Found: C, 53.84; H, 7.81; Si, 7.65; Zr, 12.40%.

## Conclusions

The catalytic activity of a wide range of metals incorporated into the structures of the MS oligomers in the curing of epoxy-diane resins was studied using fully phenyl(diethoxy)siloxy-substituted derivatives as examples. In terms of activity, the most preferable metallosiloxanes were found to be those containing Al, Hf, Nb, and in terms of cost—those containing Al, Ti, Fe<sup>II</sup>, and Zr. It was shown that the structure and functionality of the MS oligomer can serve as factors to control the formation of the MS–epoxy material since the MS ability to bind to the epoxy matrix plays an important role in the curing efficiency. This approach allows for increasing the efficiency of metal catalysis, in particular, for Zr- and Fe<sup>III</sup>-containing MS oligomers.

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