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One-step surface modification and epoxy/graphene oxide nanocomposite preparation

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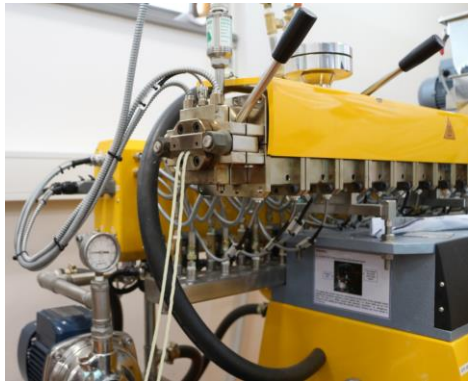

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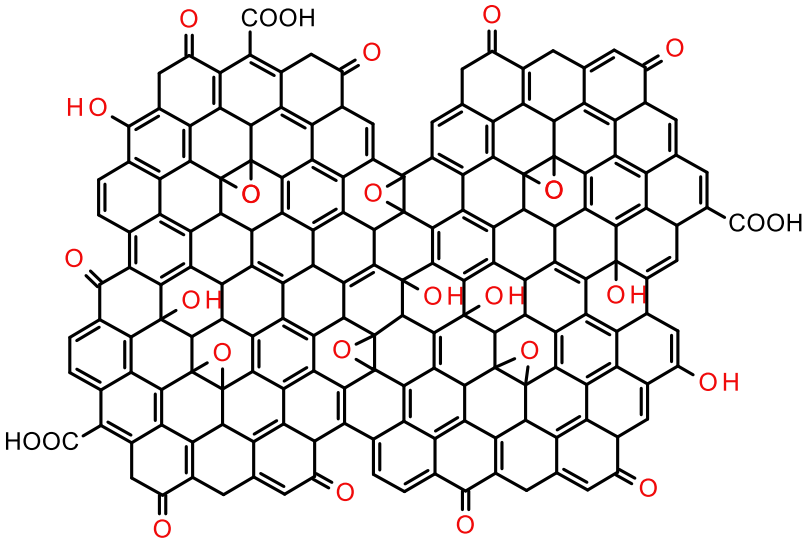
Introduction

Graphene oxide
or
Graphite oxide?



The aim of our work was threefold:

1. to prepare nanocomposites without drying the GO after its synthesis.
2. to investigate the influence of GO particle size on the properties of the epoxy nanocomposites
3. to apply selective plasma etching for GO/polymer morphology characterization

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Experimental

Materials:

Expanded graphite Sigratherm®
GFG 130 (average particle size 130 μm)
GFG1200 (average particle size 1200 μm)
are the products of SGL Group, Germany.

Epoxy resin Araldite LY 1564 and hardener Aradur 3487
are the products of Huntsman Advanced Materials.

Synthesis of GO:

Modified Hummer method.
9:1 mixture of concentrated H_2SO_4/H_3PO_4 (500 mL for GO130 and 800 ml for GO1200)
10 g of graphite flakes and 50 g of $KMnO_4$ were added.

Product was kept in a form of GO/methanol slurry.

Experimental

Nanocomposite preparation:

Concentration of GO in methanol was determined.

GO slurry was diluted with methanol and hardener (Aradur 3487) was added (GOA).

Epoxy resin LY 1564 was mixed in ratio 100:34 (resin : hardener+GOA) and cured at 100°C for 2 h in silocone mould.

Concentration of GO: 0-1%.

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XPS analysis:

Proportions of various carbon bonds together with the oxygen to carbon atomic ratio for the GO130 and GO1200.

	C-C/C=C (%)	C-O (%)	C=O (%)	COOH (%)	O/C
GO130	46	43	9	2	0.54
GO1200	41	50	7	2	0.51

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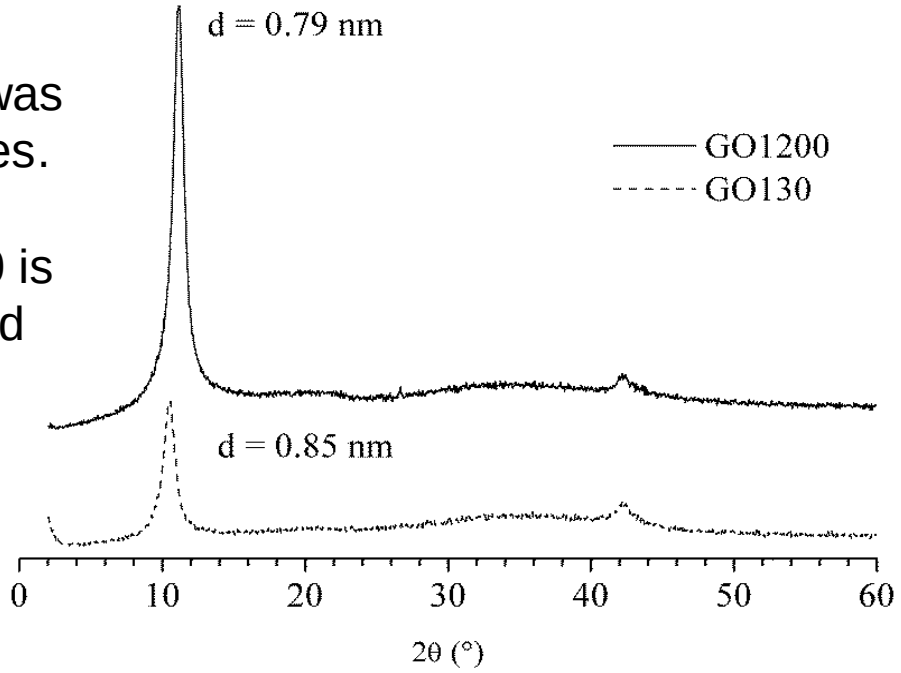
XRD:

Layered structure of the dry GO was observed by XRD for both samples.

The layer thickness of the GO130 is larger for 0.06 nm and is attributed to higher oxygen content.



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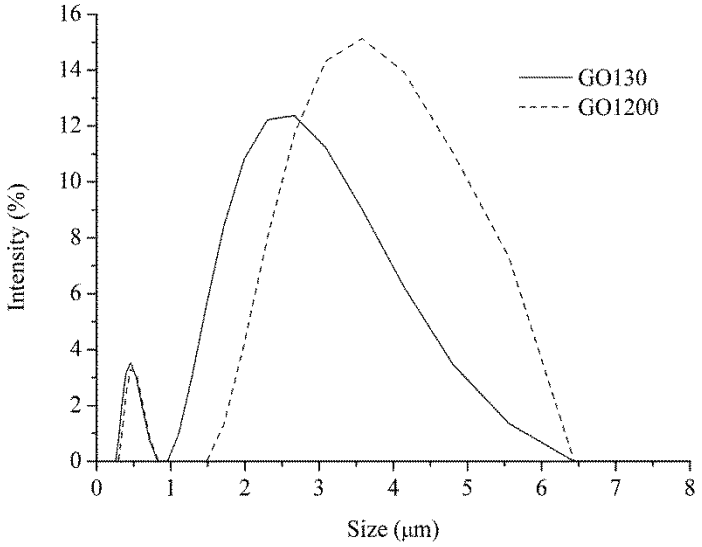
Particle size determination:

Dynamic light-scattering (DLS)
Scanning electron microscopy (SEM)



Average particle size (Z-average) and polydispersity index (PDI) of GO

	1st peak Z-av. Intensity (nm)	%	2nd peak Z-av. Intensity (μm)	%	Whole sample Z-av. (μm)	PDI	SEM (μm)
GO130	469	15.3	2.69	84.7	1.43	0.51	<80
GO1200	499	13.0	3.57	87.0	2.07	0.51	<100

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Curing of epoxy and influence on glass transition temperature

	ΔH (J/g)	T_{max} (°C)	T_g (°C)
Pure epoxy resin	-565.6	116.3	81.5
GO130-0.25 %	–566,7	116.0	85.0
GO130-0.50 %	–557.1	116.3	86.8
GO130-0.75 %	–555.1	116.4	88.3
GO130-1.00 %	–535.7	115.9	87.8
GO1200-0.25 %	–560.9	116.0	83.3
GO1200-0.50 %	–561.9	116.2	86.7
GO1200-0.75 %	–537.0	116.2	88.4
GO1200-1.00 %	–518.6	116.6	90.7

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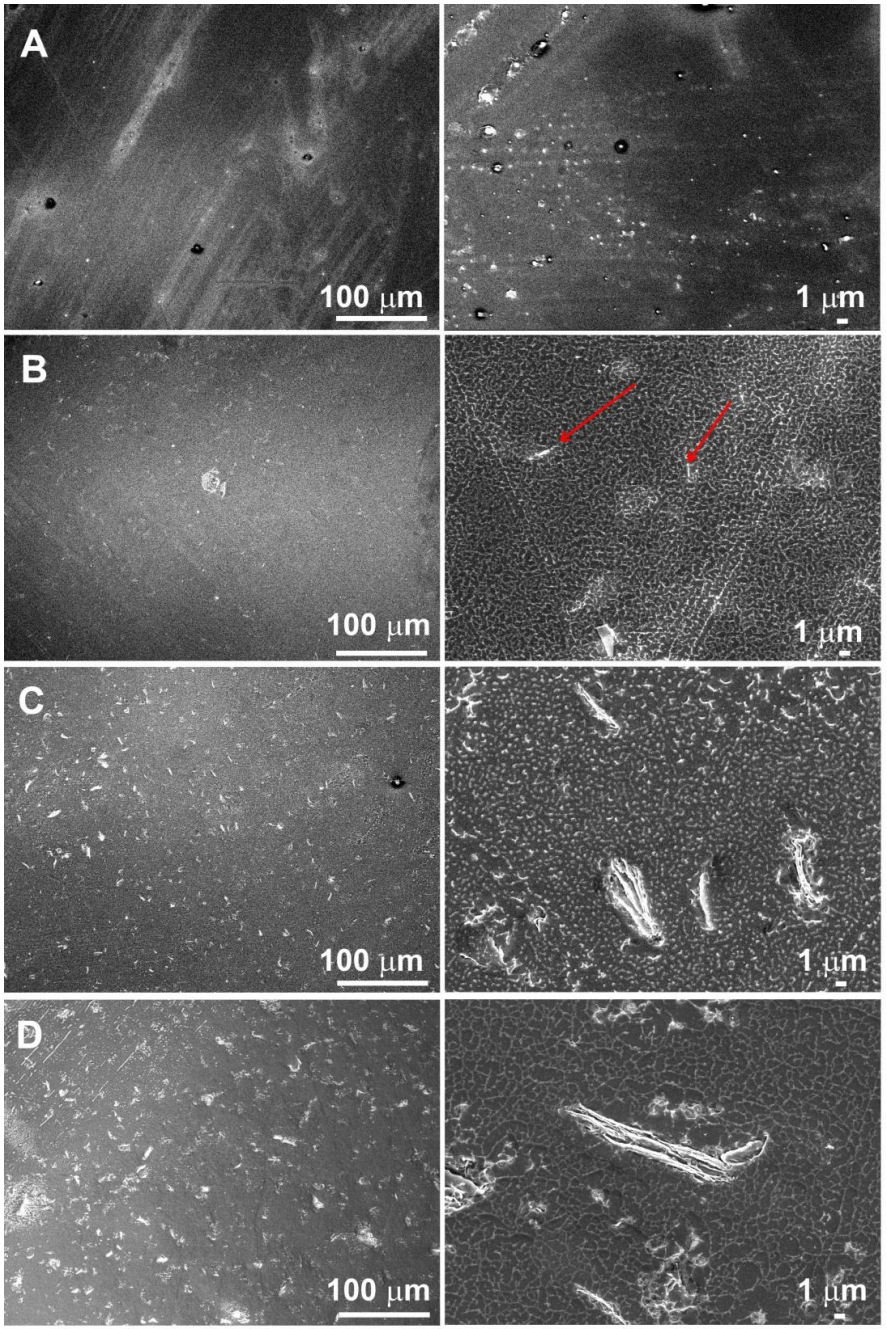
Particles distribution in epoxy

SEM images of GO1200/epoxy composite at two different magnifications: 250× (left) and 3000× (right).

- as-synthesized GO/Epoxy (A)
- for oxygen plasma-etched samples:
  (B) 2 × 3 s,
  (C) 4 × 3 s
  (D) 5 × 3 s.



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Mechanical properties

	Young's modulus (GPa)	Tensile strength (MPa)	Strain at break (%)
Pure epoxy resin	1.92 ± 0.20	60.1 ± 2.7	4.20 ± 0.29
GO130-0.25 %	2.40 ± 0.69	61.6 ± 2.5	4.61 ± 0.31
GO130-0.50 %	2.63 ± 0.42	61.9 ± 4.9	4.54 ± 0.67
GO130-0.75 %	2.68 ± 0.40	54.3 ± 4.9	3.15 ± 0.42
GO130-1.00 %	2.56 ± 0.32	48.9 ± 5.5	2.75 ± 0.47
GO1200-0.25 %	2.17 ± 0.14	67.0 ± 1.01	5.18 ± 0.25
GO1200-0.50 %	2.36 ± 0.17	66.1 ± 6.84	4.00 ± 0.78
GO1200-0.75 %	2.52 ± 0.49	63.5 ± 8.40	3.30 ± 1.00
GO1200-1.00 %	2.46 ± 0.19	66.6 ± 3.48	3.32 ± 0.49

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Conclusions

- GO can be added to curing agent as dispersion in methanol.
- Surface modified GO influence the kinetics of curing reaction.
- Young's modulus increased more when smaller GO particles were added to the epoxy resin, while the tensile strength increased for about 10 % only by the addition of larger particles.
- The optimum concentration of GO was found to be 0.25-0.50 wt.%.
- Oxygen plasma etching is valuable tool for particle size and particle size distribution determination.

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Thank you for your attention