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Fabrication of structure controllable nanocomposite with CNT aerogel by reactive infiltration of polyamide 6

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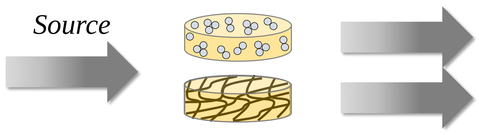
Introduction

❖ Problems in conventional nanocomposites

① **Inhomogeneous dispersion of nanoparticles → Non-uniform properties**

② **Insufficient properties (electrical/thermal conductivity, mechanical properties)**



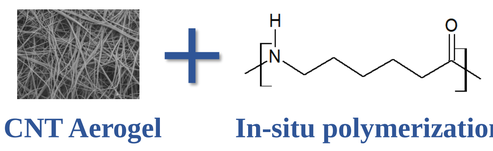
Lower conductivity due to abscission of conductive path

Higher conductivity because of continuous network structure

Objective of this research

Introduction of **controllable nanostructure** with a remarkable performance

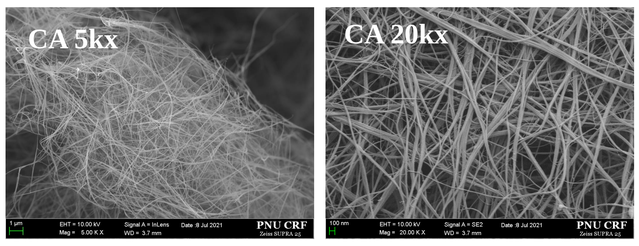
Breakthrough solution in nanocomposites



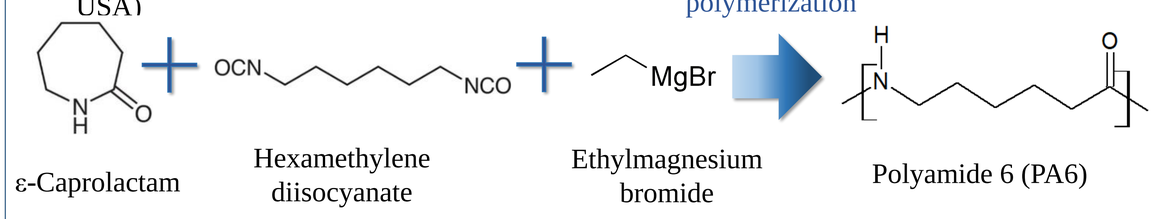
Experiment

❖ **Materials**

- CNT aerogel (CA, density 0.05~0.1 g/cm^3)
- ε-Caprolactam (Monomer, melting point 68˚C, Sigma Aldrich, USA)
- Ethylmagnesium bromide solution (Catalyst, 3.0 M in diethyl ether, Sigma Aldrich, USA)
- Hexamethylene diisocyanate (Activator, ≥98.0%, Sigma Aldrich, USA)



< The images of nano-pores in CNT aerogel >

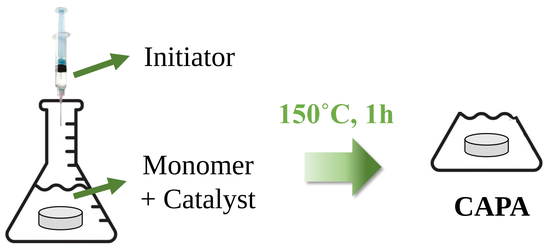


< Schematic figure of reactive in-situ polymerization of PA6 >

Experiment

❖ CNT aerogel/Polyamide 6 composites (CAPA) were fabricated by dipping and T-RTM (Thermoplastic resin transfer molding) method using reactive in-situ polymerization.

Dipping

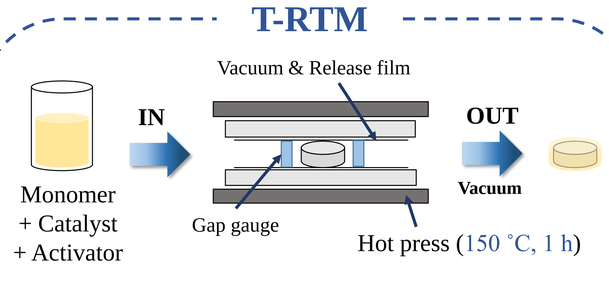


✓ Simple and easy method to fabricate

✓ Difficulty of composite shape control

✓ Low quality of composites (pore formed)

T-RTM



✓ Fabrication of composite with dense structure using vacuum pressure (easy impregnation)

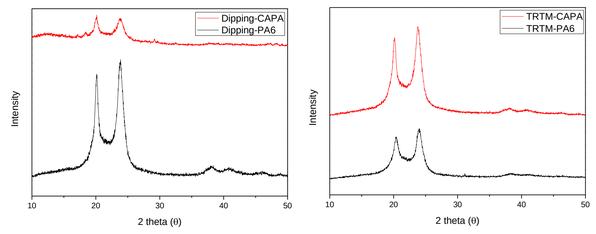
✓ Shape controllable composite

✓ Process for massive composites

Results & Discussion

❖ Measurement of crystallite size of CAPA by dipping and T-RTM method

- **XRD**



$$D = \frac{k\lambda}{\beta \cos\theta}$$

< Scherrer equation >

- D : crystallite size
- β : FWHM
- λ : wavelength of the X-ray
- k : Scherrer constant

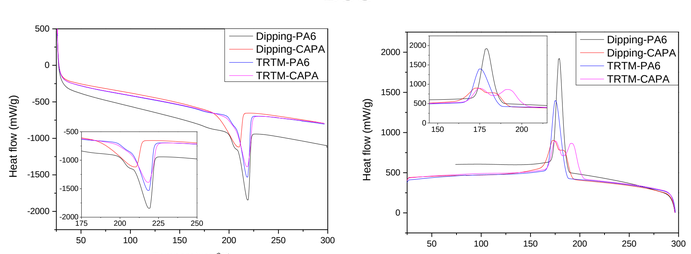
S.N.	FWHM	Crystallite Size (nm)
PA6	3.21	2.44
	1.61	4.83
CAPA	2.71	2.88
	1.86	4.18

- As a results of XRD, crystallite size of CAPA was same as neat PA6, indicating that 3D network structure of CA did not interrupt polymerization of PA6.

Results & Discussion

❖ Measurement of thermal properties (DSC) of CAPA by dipping and T-RTM method

- **DSC**

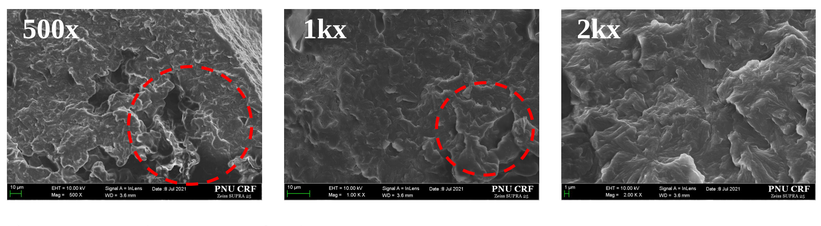


	T_m (˚C)	T_c (˚C)
Dipping-PA6	218.2/205.9	178.8
Dipping-CAPA	218.2/208.6	172.6/183.4
TRTM-PA6	218.2	175.1
TRTM-CAPA	218.2	175.1/191.6

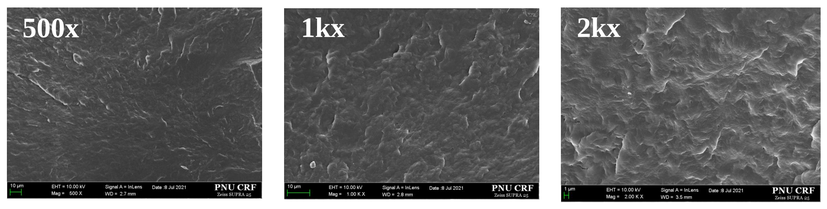
- Slow annealing in oil bath and unpolymerized PA6 with dipping process led to two T_m peaks.
- Two T_c peaks of CAPA by both method occurred because CNT acts as a nucleation agent for forming crystalline structure of PA6.

❖ Morphology characterization of CAPAs using different process (FE-SEM)

➢ **Dipping (cross section)**

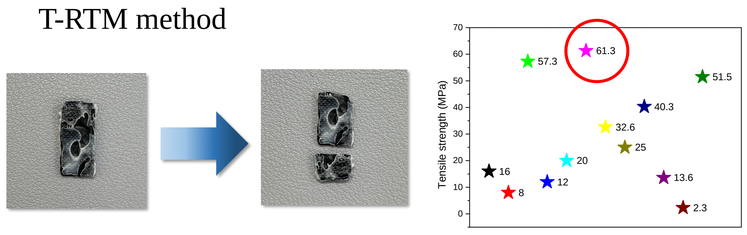


➢ **T-RTM (cross section)**



- The FE-SEM images of composite were characterized for comparing morphology by different process.
- Nanocomposite using dipping process has many void while homogeneous and uniform surface was observed with T-RTM process. This is because T-RTM method uses vacuum pressure which is stronger than dipping.

❖ Characterization of mechanical properties of CAPA by T-RTM method



< Comparison of mechanical property of composites with 1wt% of CNT>

No.	Tensile strength (MPa)	Material	Reference
1	61.3	$CNT_{aerogel}$/PA6	**This work**
2	20	CNT/PA6	Materials Chemistry and Physics 117 (2009) 313–320
3	25	CNT/PA6	CompositesScienceandTechnology72(2012)1918–1923
4	40.3	CNT/PA6	Macromolecules 2004, 37, 7214-7222
5	32.6	CNT/PA6	Journal of Polymer Research 18 (2011): 2055-2060
6	13.60	$CNT\&GO_{aerogel}$/PS	Composites Science and Technology 195 (2020) 108191
7	2.3	$CNT_{aerogel}$/PDMS	Composites Part A: Applied Science and Manufacturing, 90, 678-686.
8	57.3	CNT-OH/PA6	Composites Research 32.6 (2019): 375-381.
9	51.5	CNT-COOH/PA6	Macromolecules 37.2 (2004): 256-259.

Conclusion

1. In this study, We successfully obtained PA6 impregnated CNT aerogel composites through reactive in-situ polymerization by dipping and T-RTM methods.
2. Crystallite size of neat PA6 and CAPA was measured by Scherrer equation, which was indicated that stereoscopic structure of CA do not restrict to polymerize.
3. DSC results showed thermal properties of neat PA6 and CAPA. There were two peaks of T_m and T_c whose mechanisms were revealed.
4. The FE-SEM images of CAPAs showed that the difference between two processes, indicating that T-RTM was better way to fabricate CAPA than dipping.
5. It was indicated that mechanical performance of CAPA is much better than other composites which used CNT nanoparticles as usual.

❖ **Acknowledgement**

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