



### Development of a novel high-performance selflubricating micro/nano mould based on 2D material nanocomposite

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#### BACKGROUND









Mismatch of thermal expansion coefficient: Nickel:  $K=13 \times 10^{-6} 1/K$ , PMMA:  $K=70 \times 10^{-6} 1/K$ 

#### BACKGROUND







### BACKGROUND

Distortion and damage of surface micro patterns

500µm

[2]





(C)



#### MOTIVATION



Coatings	Friction coefficient	Coating methods	Advantages	Disadvantages
Fluoropolymer coatings (PTFE, PFPE, FEP)	0.05~0.1	PVD, CVD,	Hydrophobic surface	<ul><li>Poor wear resistance</li><li>Lower service temperature</li></ul>
Cubic boron nitride (c- BN) coating	<ul> <li>Dry: 0.2~0.6</li> <li>Humid: &lt;0.1</li> </ul>	CVD	<ul><li>High service temperature</li><li>High oxidation resistance</li><li>High wear resistance</li></ul>	<ul><li>Poor adhesion to the substrate</li><li>High residual stress</li><li>Expensive coating facility</li></ul>
Self-assembled monolayer coatings (SAM)	0.07~0.1	Dip coating Molecular vapor deposition	Low-cost process	<ul><li>Low wear resistance</li><li>Low thermal stability</li></ul>
Diamond-like carbon (DLC)	<ul> <li>Dry: 0.001~0.05</li> <li>Humid: 0.2~0.3</li> </ul>	Sputtering, thermal evaporation, PECVD	<ul><li>High hardness</li><li>Low friction</li></ul>	<ul> <li>High internal stress</li> <li>Poor adhesion to substrate</li> <li>Restriction in thickness</li> <li>Expensive to CVD coating</li> </ul>
Graphene/ Graphene oxide	<ul><li>Dry: 0.15~0.2</li><li>Humid: 0.15~0.2</li></ul>	CVD, Chemical and mechanical exfoliation	<ul><li>Stable coefficient of friction;</li><li>Good filler material</li></ul>	<ul> <li>Difficult for large-area coatings</li> <li>0.1-0.2 nm coating thickness</li> </ul>
MoS <sub>2</sub> and WS <sub>2</sub>	<ul> <li>Dry: 0.02~0.06</li> <li>Humid: 0.15~0.25</li> </ul>	Sputtering, thermal evaporation, CVD	High temperature	<ul> <li>Lower wear resistance of MoS<sub>2</sub></li> <li>WS<sub>2</sub> loses its lubricating properties in humid environments</li> </ul>







#### MOTIVATION

- Image: Second secon
  - ✓ Mechanical properties: *High microhardness and longer tool life*
  - ✓ Tribological properties: *Low friction and adhesion*
  - ✓ Surface roughness: *Less than 100nm*
  - ✓ Dimensional accuracy: *high dimensional accuracy*



#### **2D MATERIALS SELECTION**





AFM tip-based technology



Experimental methods:

 ✓ Types of 2D material: *GO*; *MoS*<sub>2</sub>; *WS*<sub>2</sub>

 ✓ Concentration of 2D material: *GO* & *MoS*<sub>2</sub>: 0.1, 0.2, 0.5, 1.0 g/L *WS*<sub>2</sub>: 0.1, 0.14, 0.2, 0.5 g/L

#### ELECTROLYTE PREPARATION





Structures of few-layered  $MoS_2/WS_2$  (a) and GO (b), respectively; preparation of electroforming solution containing 2D materials dispersion (c).

Electroforming experimental setup for the fabrication of 2D materials reinforced nickel moulds (a) and related demoulding method for releasing electroformed moulds (b).

#### SURFACE MORPHOLOGY



Nickel/GO composite moulds Nickel/MoS<sub>2</sub> composite moulds Nickel/WS<sub>2</sub> composite moulds 0.2 g/L GO 0.1 g/L GO 0.14 g/L WS2 0.1 g/L WS2 0.1 g/L WS2 0.14 g/L WS2 GO aggregates 0.5 g/L GO 1.0 g/L GO 0.2 g/L WS2 0.5 g/L WS2 0.2 g/L WS2 0.5 g/L WS2 Large S<sub>2</sub> particle Large WS<sub>2</sub> particles Large WS<sub>2</sub> particles Large WS<sub>2</sub> particles arge GO particles

## CROSS-SECTION MORPHOLOGY OF NICKEL/ GO MOULDS





Cross-section morphology of electroformed nickel/GO composite moulds, fabricated in nickel sulfamate bath with various concentrations of GO: (a) 0.1 g/L GO; (b) 0.2 g/L GO; (c) 0.5 g/L GO; (d) 1.0 g/L GO; and corresponding GO content in the deposit (f), where the content of GO is determined by C content in the deposit.

# CROSS-SECTION MORPHOLOGY OF NICKEL/M<sub>O</sub>S<sub>2</sub> MOULDS





Cross-section morphology of electroformed nickel/MoS<sub>2</sub> composite moulds (a-e), fabricated in nickel sulfamate bath with various concentrations of MoS<sub>2</sub>: (a)  $0.1 \text{ g/L MoS}_2$ ; (b)  $0.2 \text{ g/L MoS}_2$ ; (c)  $0.5 \text{ g/L MoS}_2$ ; (d)  $1.0 \text{ g/L MoS}_2$ ; and corresponding MoS<sub>2</sub> content in the deposit (f), where the content of MoS<sub>2</sub> is determined by Mo content in the deposit.

# CROSS-SECTION MORPHOLOGY OF NICKEL/WS<sub>2</sub> MOULDS





Cross-section morphology of electroformed nickel/WS<sub>2</sub> composite moulds (a-e), fabricated in nickel sulfamate bath with various concentrations of WS<sub>2</sub>: (a)  $0.1 \text{ g/L WS}_2$ ; (b)  $0.14 \text{ g/L WS}_2$ ; (c)  $0.2 \text{ g/L WS}_2$ ; (d)  $0.5 \text{ g/L WS}_2$ ; and corresponding WS<sub>2</sub> content in the deposit (f), where the content of WS<sub>2</sub> is determined by W content in the deposit.

#### **MICROSTRUCTURE ANALYSIS**





XRD patterns of electroformed pure nickel mould and nickel composite moulds fabricated in nickel sulfamate bath with various concentrations of 2D materials: (a) nickel/GO composite moulds; (b) nickel/MoS<sub>2</sub> composite moulds; (c) nickel/WS<sub>2</sub> composite moulds.

Sherrer's equation • L is the average crystallite size,  $\beta$  is the full-width half (FWHM) maxima.  $L = \left(\frac{K\lambda}{\beta \,\cos\theta}\right)$ 

• *FWHM* is used to calculate the crystallite size.

*FWHM broadening* means the *refinement* of crystallite size.

□ FWHM is broadening with *increasing 2D materials concentration* and the cases of *WS*<sub>2</sub> are more *prominent*.

#### MICROHARDNESS AND CRYSTALLITE SIZE





#### SURFACE ROUGHNESS AND WETTABILITY ANALYSIS





Surface roughness increases because of embedding of 2D materials

Wettability varies with 2D materials concentration and surface topography from hydrophilic to hydrophobic

Concentration of 2D materials in nickel sulfamate bath (g/L)

#### FRICTION COEFFICIENT





#### *Friction stage:* wear morphology



**Figure 16.** Wear morphology of pure nickel and nickel/2D material composite moulds during the initial friction stage: (a) pure nickel; (b) 0.5 g/L GO; (c) 1.0 g/L MoS<sub>2</sub>; (d) 0.14 g/L WS<sub>2</sub>. Note that selected concentrations of 2D materials are optimal to their respective lowest coefficient of friction.

#### WEAR MORPHOLOGY OF NICKEL/ GO MOULDS



E M B

# WEAR MORPHOLOGY OF NICKEL/M<sub>O</sub>S<sub>2</sub> Montactures MOULDS





#### WEAR MORPHOLOGY OF NICKEL/WS<sub>2</sub> MOULDS



E M B

## SELF-LUBRICATING MECHANISM OF NICKEL/2D MATERIALS MOULD





Schematic illustration of wear mechanisms of pure nickel mould (a); self-lubricating mechanisms of 2D materials enhanced nickel moulds (b) and (c).

### CONCLUSION



- *A low concentration of 2D materials* is recommended to be used to achieve homogenous distribution in the electroformed moulds.
- The role of GO and MoS<sub>2</sub> in enhancing the performance of nickel mould tools is reflected in mechanical properties and tribological properties, respectively, whereas WS<sub>2</sub> is both compatible and more significant.
- The incorporation of 2D materials not only modulates the nickel matrix grains growth but also easily forms *self-lubricating transfer film*, especially for  $WS_2/MoS_2$ .
- The main wear mechanism is altered from *abrasive-dominated wear* for pure nickel mould to *adhesion-dominated wear* for nickel/2D materials composite moulds.
- Maximum microhardness of ~660 HV is achieved from 0.5 g/L WS<sub>2</sub>, indicating a 3.67 *times* increase compared with pure nickel mould.
- A low concentration (0.14 g/L) of  $WS_2$  shows the lowest COF, where the COF in the initial friction stage is 0.08, implying a decrease of 42.8% and 27 times increase in a lifetime, compared with pure nickel mould.







### Thank you for your kind attention!





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