Evaluation of Mechanical Properties of Electroplated Nickel Using Micro-Compression Test

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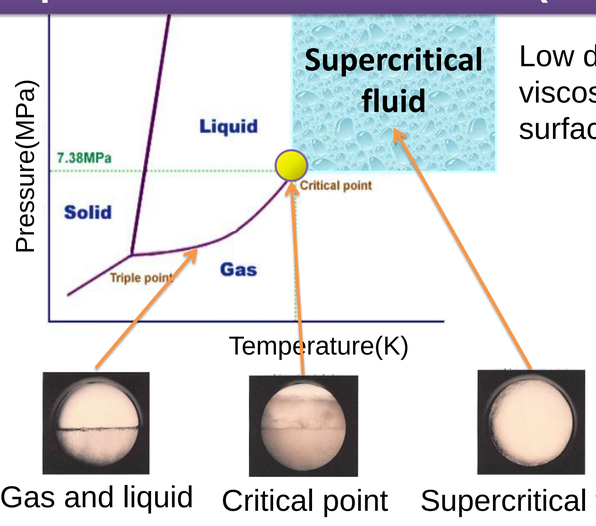
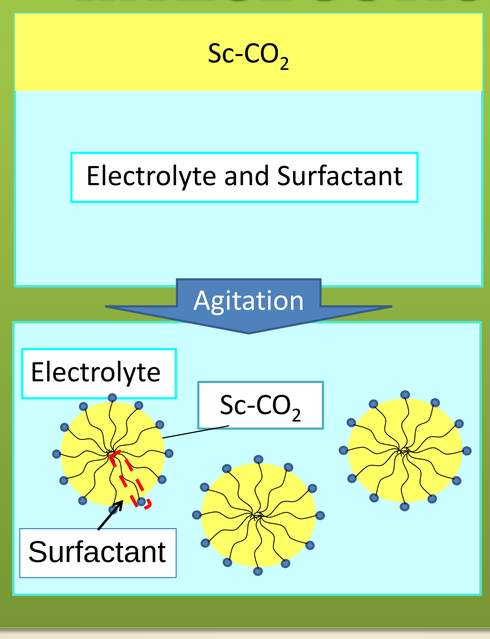
INTRODUCTION

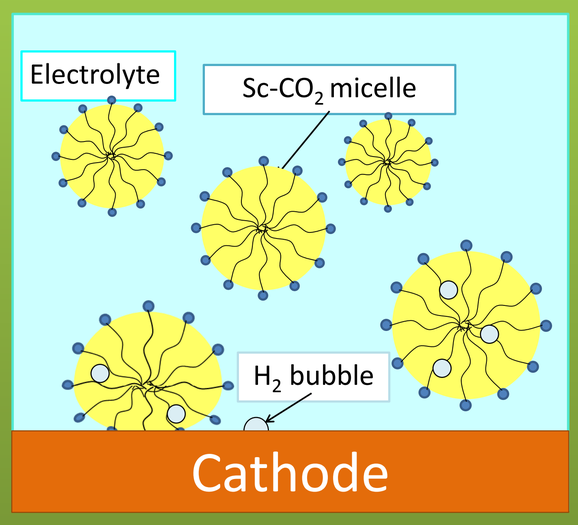
Supercritical Carbon Dioxide (Sc-CO_2)



Low density, viscosity, and surface tension



Electroplating With Sc-CO_2 Emulsion (ESCE)



✓Increased desorption of H_2 bubble from cathode

Void and defect free

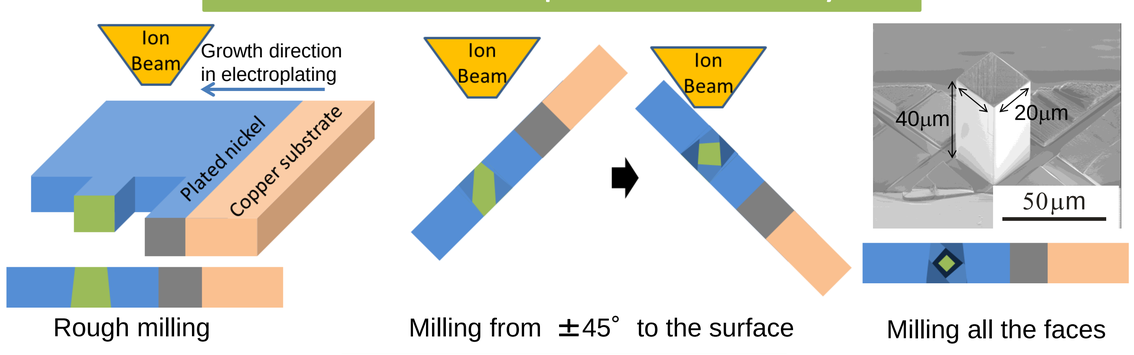
✓Periodic on/off at the surface of the cathode

Grain refinement

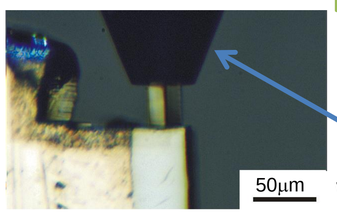
Enhanced mechanical properties

EXPERIMENTAL PROCEDURE

Fabrication of Compression Pillar By FIB



Micro-Compression Test



Control	Displacement rate	Load resolution
Displacement control	0.1 [μm/s]	10 [μN]

Materials
Substrate
➢Cathode : Cu substrates ➢Anode : Ni substrates
Additive Free Watts Bath
➢ $NiSO_4 \cdot 6H_2O$ (300 g/l) ➢ $NiCl_2 \cdot 6H_2O$ (50 g/l) ➢ H_3BO_3(50 g/l)
Surfactant
➢ polyoxyethylene lauryl ether ($C_{12}H_{25}(OCH_2CH_2)_{15}OH$)

Pretreatment

1. Degreasing, 10 wt% Ace clean solution for 1min
2. Remove oxide layer, 10 wt% HCl solution for 10 sec

Electroplating With Sc-CO_2

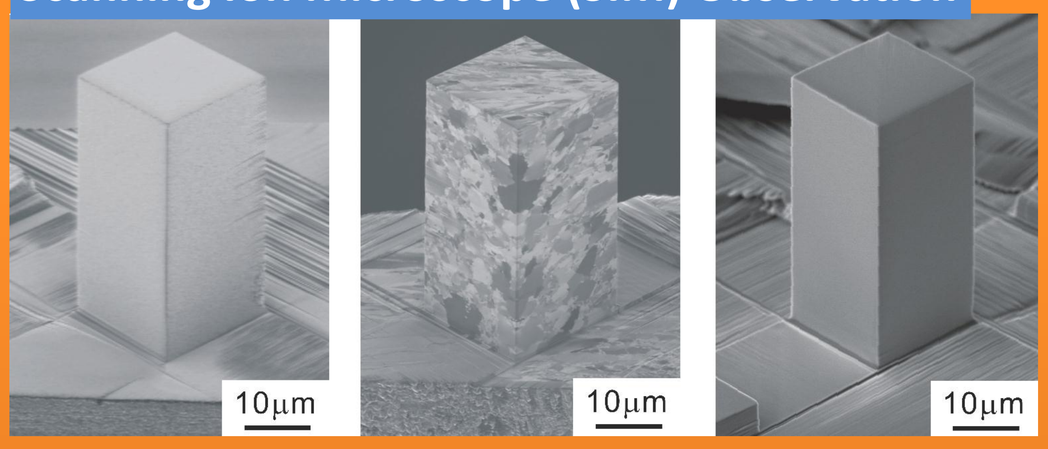
Plating Condition

	ESCE	High Pressure Electroplating (HPE)
Temperature	323 K	
CO_2 vol%	20 vol%	
Current Density	2 A/dm^2	
Pressure	15 MPa	6 MPa
Agitation	On	Off

To prove how much would the emulsified Sc-CO_2 affect the structure and properties

RESULTS & DISCUSSION

Scanning Ion Microscope (SIM) Observation

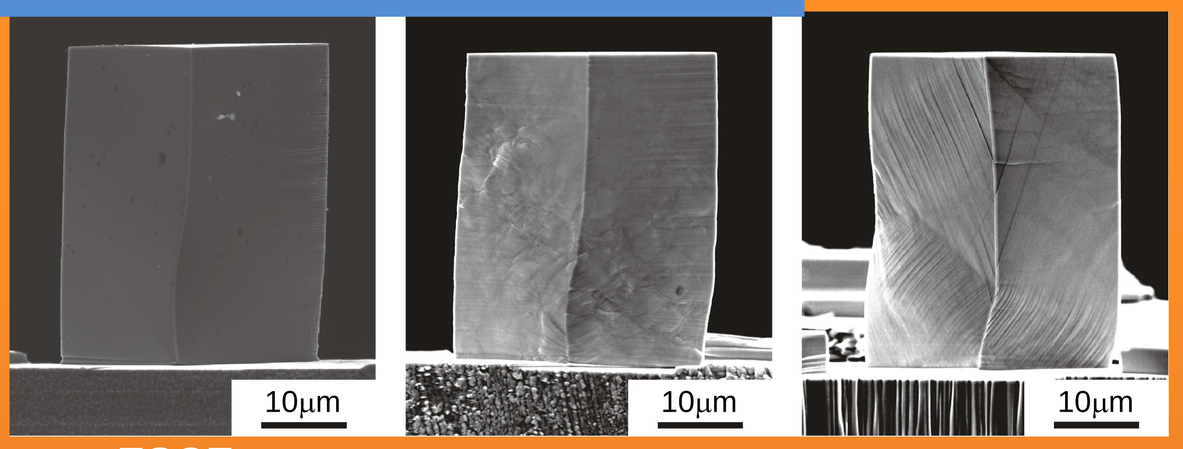


ESCE HPE Single Cristal (SC)

Image contrast represents difference in crystal orientation

ESCE	Grains are finer than the SIM resolution
HPE	Columnar grains with diameter of 2 μm
SC	Single crystal

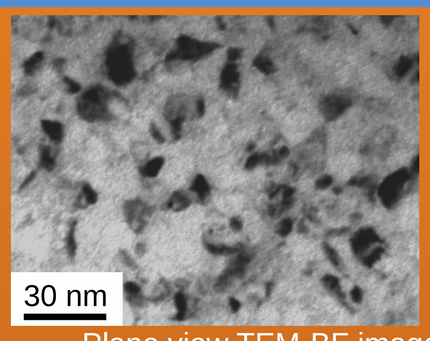
SEM Observation After Deformation



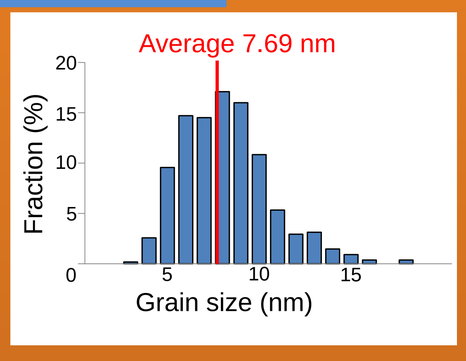
ESCE HPE SC

ESCE	No crack or slip band is observed
HPE	Slip line are visible in individual grains
SC	Several different slip lines are visible

TEM Observations of ESCE Nickel

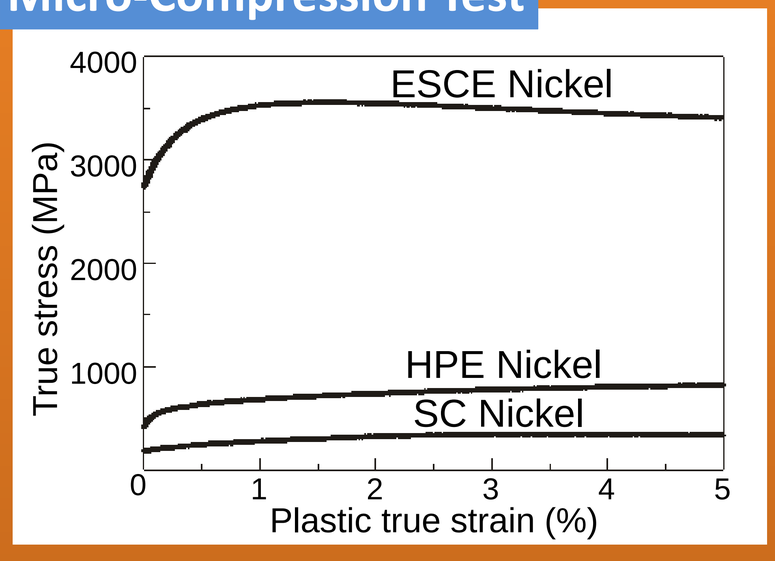


Plane view TEM-BF image



Equiaxed nanocrystalline nickel were obtained by periodic on/off characteristics in ESCE

Micro-Compression Test



Yield strength of ESCE Nickel is 10 times higher than SC & 5 times higher than HPE

- Compressive strength exceeds 3500 MPa in micro-compression
- Cracks or failure are absent up to 9 % of permanent strain

ESCE is a good candidate to improve mechanical properties of plated film

CONCLUSIONS

- **Due to the periodic on/off characteristics in ESCE by bouncing micelles on the cathode, grain size decreased to 8 nm.**
- **As grain size decreased from 2 μm to 8 nm, yield stress increased by a factor of around five. And maximum stress for ESCE nickel reaches 3.5 GPa without any crack or failure up to 9% of permanent strain.**