Effect of Annealing on Mechanical Properties of Nickel Film Electrodeposited Using Supercritical CO_2 Emulsion Evaluated by Micro-Compression Test

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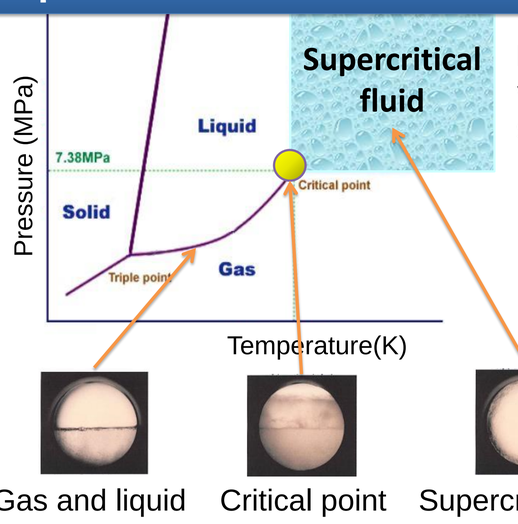
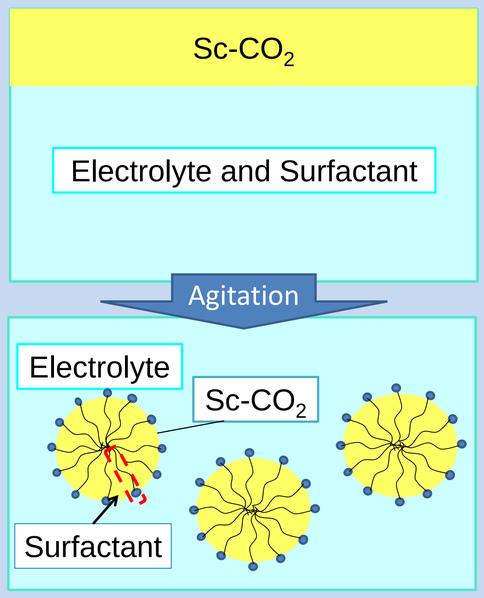
INTRODUCTION

Supercritical Carbon Dioxide (Sc-CO_2)

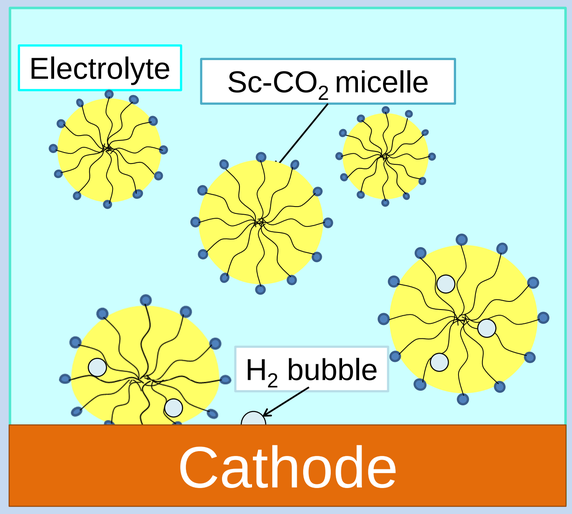


Low density, viscosity, and surface tension.

Use supercritical CO_2 for electroplating



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

Improved strength

Possible application for MEMS components.

EXPERIMENTAL PROCEDURE

Electroplating With Sc-CO_2

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 ($C_{12}H_{25}(OCH_2CH_2)_{15}OH$)

Plating conditions	
Temperature	323 K
CO_2 vol%	20 vol%
Current density	2 A/dm^2
Pressure	15MPa

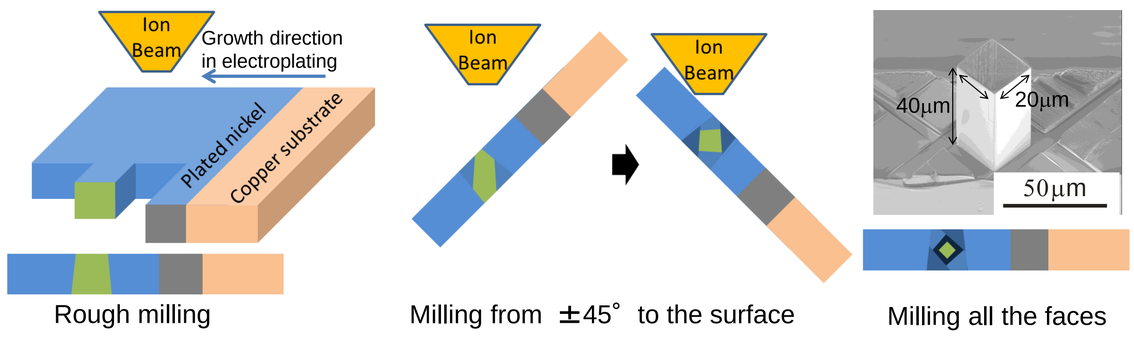
Annealing of plated nickel

In vacuum for 2 hours at 150, 200, 300, 400℃

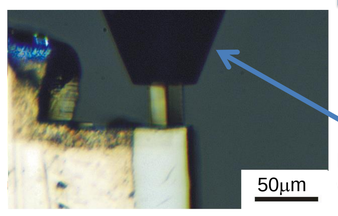
Structural characterization
- ➢ Transmission electron backscattered diffraction (t-EBSD)
- ➢ SEM-STEM

Mechanical characterization
- ➢ Micro-compression test

Fabrication of Compression Pillar by FIB



Micro-Compression Test



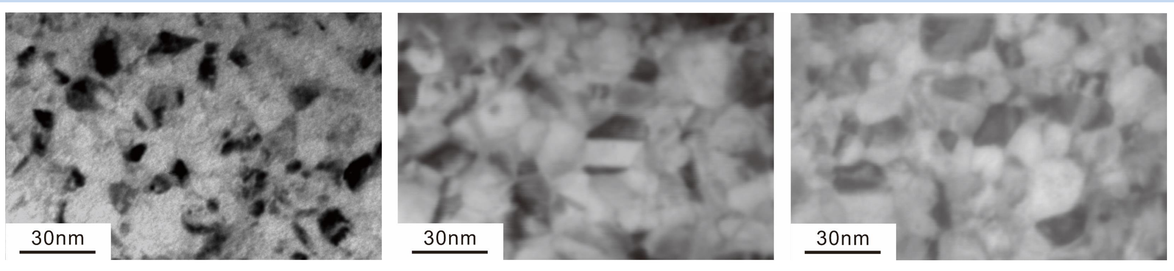
Flat diamond indenter with tip diameter of 50 μm

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

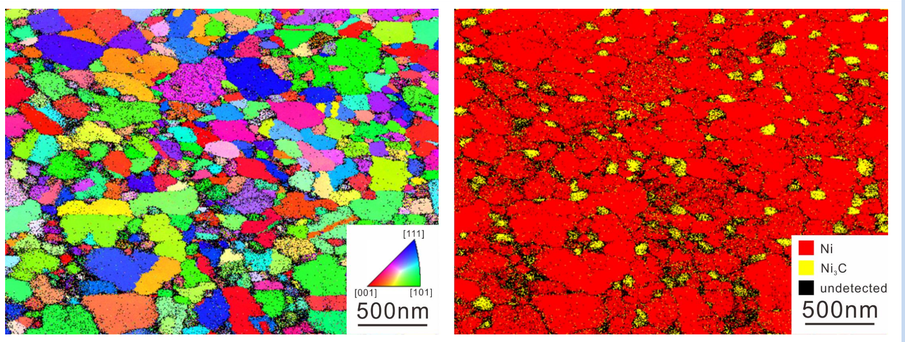
RESULTS & DISCUSSION

Effect of Annealing

As deposited | 150℃ annealed | 200℃ annealed

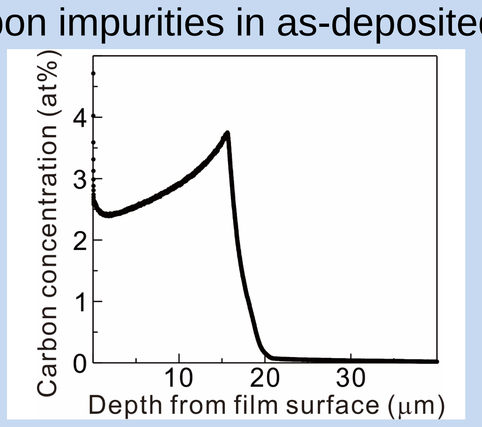


t-EBSD 300℃ annealed



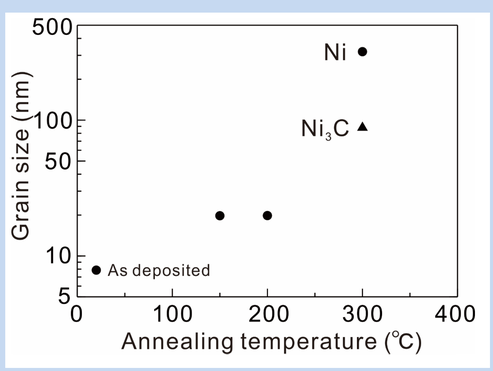
Grain growth and Ni_3C precipitation observed at 300℃ annealing

Carbon impurities in as-deposited Ni



Carbon impurities from CO_2 produce fine nickel carbide on grain boundary

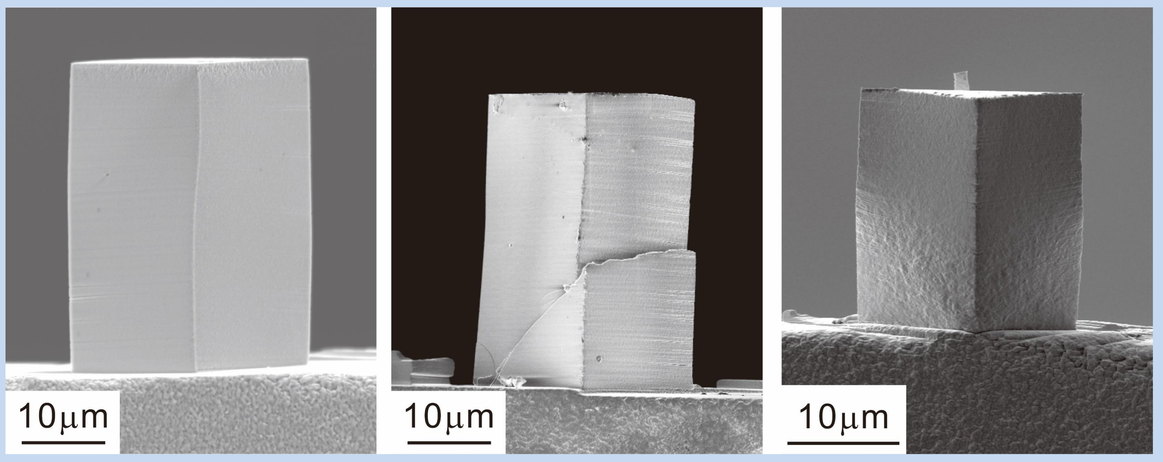
Grain size



Grain growth at 300℃ results in the 300 nm grains with high fractions of twin boundaries

Mechanical Properties

As deposited | 200℃ annealed | 300℃ annealed



➢ 200℃ pillar indicate brittle fracture and others deformed by broad shear banding

Micro-compression test

Hall-Petch plot

➢ Yield strength increased by low temperature annealing.
➢ Carbon may rearranged on grain boundary which improve boundary strength and form intermetallic on grain boundary.

CONCLUSIONS

Low temperature annealing of nanocrystalline nickel make material brittle and increase yield strength.
Carbon impurity found in nickel film may concentrate on grain boundary by low temperature annealing and produce carbide at grain boundaries which suppress grain growth.