P3-11-Wd

Mechanical Properties of Sn Electrodeposited in Supercritical CO_2 Emulsion Using Micro-Compression Test

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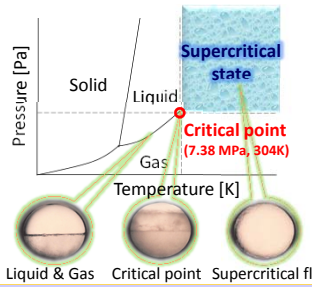
Introduction

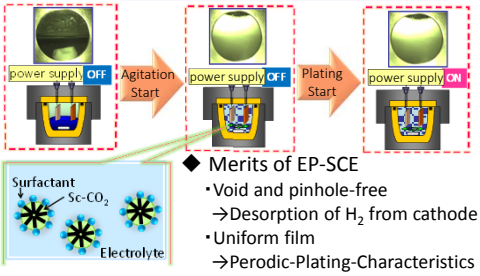
Supercritical carbon dioxide (sc-CO_2)



◆ Feature of sc-CO_2
- low viscosity (high diffusivity)
- low density
- miscibility with H_2

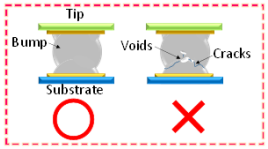
To conduct electrochemical reactions, surfactant is added to form sc-CO_2 emulsified electrolyte

Electroplating with sc-CO_2 emulsion (EP-SCE)



◆ Merits of EP-SCE
- Void and pinhole-free
  →Desorption of H_2 from cathode
- Uniform film
  →Perodic-Plating-Characteristics

Use of Sn in load bearing component

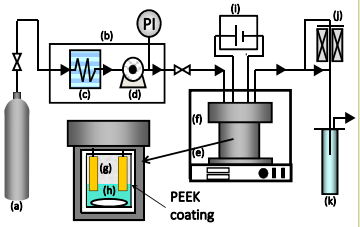


Reliability & Long life

Mechanical property

Experimental

High pressure apparatus



(a)CO_2 gas tank, (b)CO_2 liquidization unit
(c)Liquidizatrion unit, (d)High pressure pump
(e)Thermal bath, (f)Reaction cell (SUS316L) with PEEK coating inside, (g)Electrodes
(h)Cross stirrer, (i)Power supply
(j)Back pressure regulator, (k)trap

Materials

Electrodes
- cathode : Ni
- anode : Sn

Electrolyte
- $SnCl_2 \cdot 2H_2O$ (0.22 mol/L)
- $HOC(COONH_4)(CH_2COONH_4)_2$ (0.31mol/L)
- HCl (0.015 mol/L)
- CO_2 (20 vol.%)

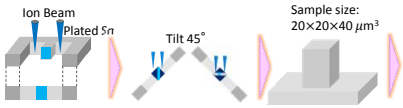
Surfactant (EP-SCE)
- polyoxyethylene lauryl ether ($C_{12}H_{25}(OCH_2CH_2)_{15}OH$) 0.2 vol.% with electrolyte

Conditions

- Pressure : atomosphere (CONV) 15 MPa (EP-SCE)
- Temperature : 343 K
- Current density : 8.7 , 17.3 mA/cm²
- Plating time : 150, 75min (respectively)

Theoretical film thickness , about 65 μm

Micro-compression test



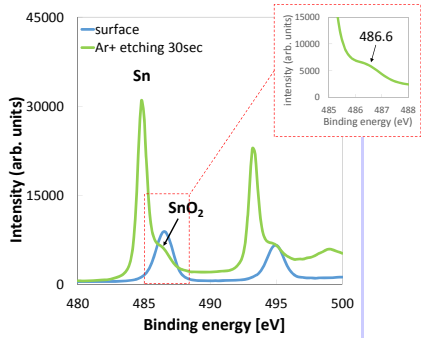
- Displacement rate : 0.1 μm/s
- Load resolution : 10 μm
- Displacement resolution : 10 μm

Fabricated by FIB → Micro-compression test

Results and discussion

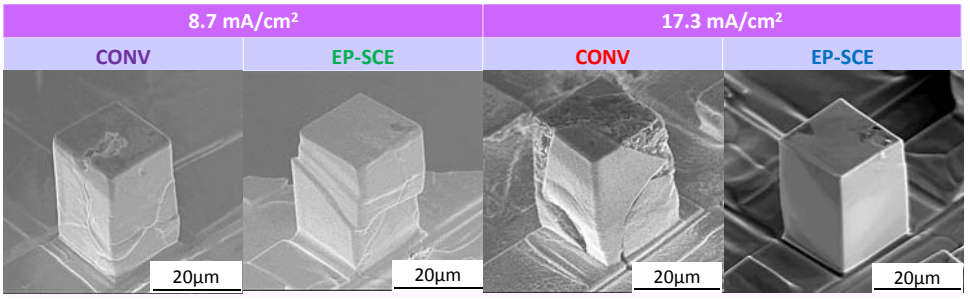
Composition analyzed by XPS

17.3 mA/cm²

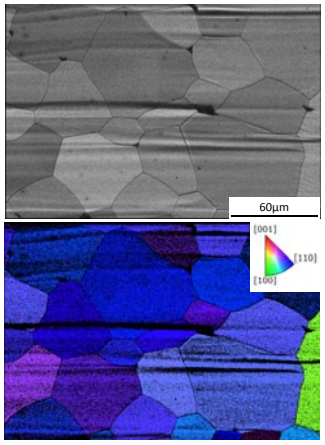


- SnO_2 is in the film by CONV.
- The film by EP-SCE consists of pure Sn.

Deformed Sn pillars

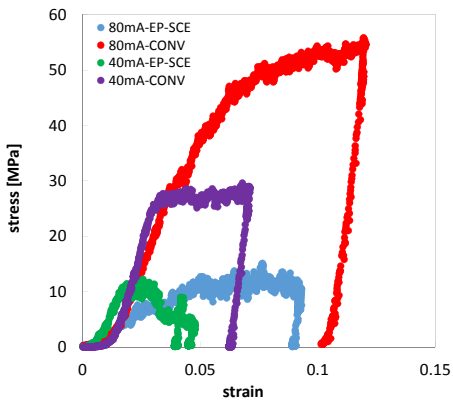


Grain size of EP-SCE by EBSD



Grain size ≅ 60 μm

Mechanical strength



The yield strength by CONV is higher than that by EP-SCE.
→SnO_2 is in the pillar by CONV.

Conclusions

- Grain size of EP-SCE is about 60 μm.
- SnO_2 is in the film by CONV.
- The yield strength by CONV is higher than that by EP-SCE.

References

[1]T.F.M. Chang, M. Sone, Surf. Coat. Technol. 205 (2011) 3890-3899
[2]M. Tanabe, T.F.M. Chang, H. Hosoda, T. Sato, M. Sone, ECS Electrochem. Lett., 3 (2014) D44-D45

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