

Validation of the Dynamic Recrystallization (DRX) Mechanism for Whisker and Hillock Growth in Thin Films

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Outline

◆ Introduction

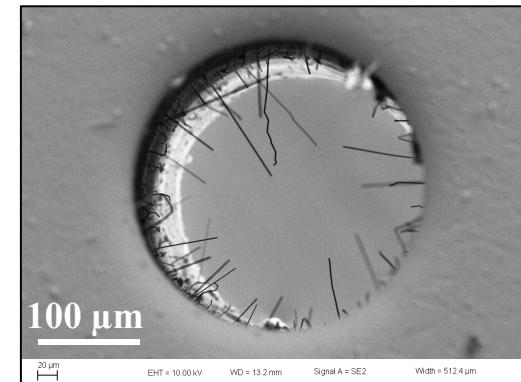
- Whiskers in metal and alloys
- Dynamic recrystallization (DRX) – (very) brief description
- DRX applied to whisker and hillock formation

◆ Creep Data of Sn

- Bi-modal deformation kinetics

◆ Validation of the DRX-based model

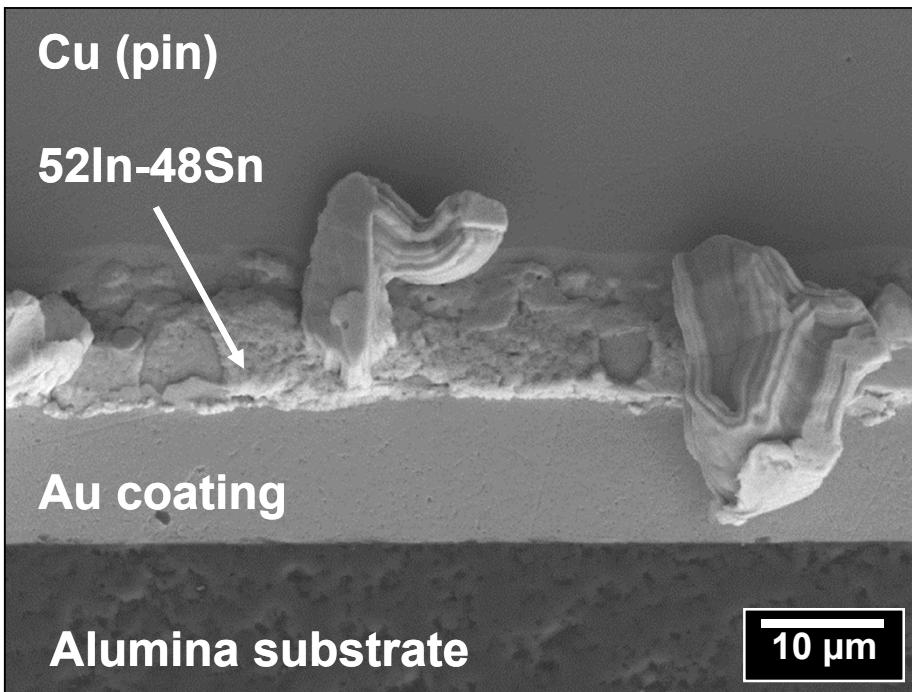
- Creep experiments: Sn on Si wafers
- Data: Long whiskers, hillocks and depleted zones
- Analysis ... DRX driving force
... Thin film effects



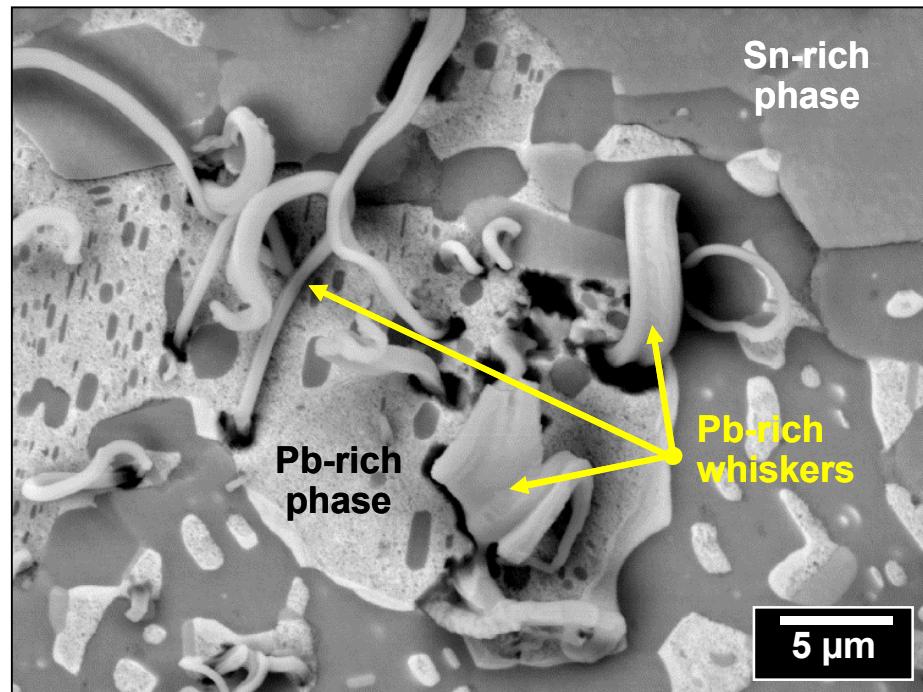
Immersion Sn – plated through hole

Introduction

- ◆ The formation of whiskers occurs across a wide spectrum of metals – Cd, Sn, In, Zn, **Au**, Pb – and alloys – In-Sn and Sn-Pb.
- ◆ Whisker formation is a relatively general occurrence; *however, Sn is particularly prone to this phenomenon.*



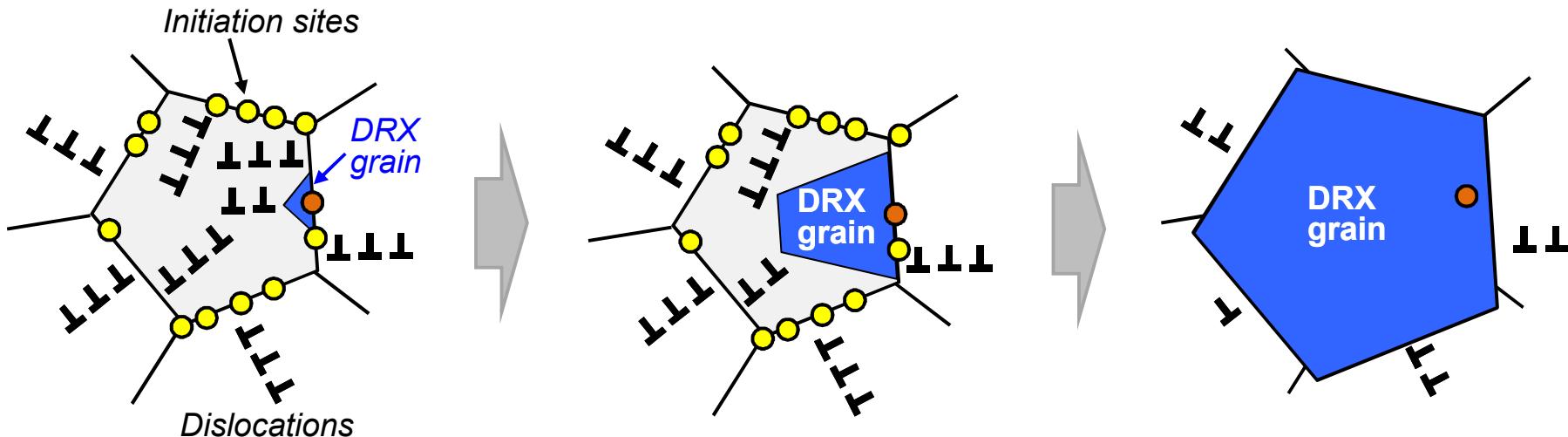
Whisker growth from In- and Sn-rich phase grains.



Pb whiskers grow from Sn-Pb solder in a metallographic cross section.

Introduction

- ◆ **Functional definition of dynamic recrystallization (DRX):**
 - **Recrystallization – new grain formation-plus-grain growth – that initiates sooner than during static recrystallization (SRX) due to the additional driving force provided by the strain energy.**



Recrystallized grain formation (initiation) at the orange site.

The elimination of strain energy provides the driving force for DRX grain growth.

DRX grain growth ends when the strain energy is lost as the driving force.

Introduction

- ◆ Dynamic recrystallization (DRX) takes place when the strain, ϵ , exceeds the **critical strain value**, ϵ_c :

$$\epsilon > \epsilon_c$$

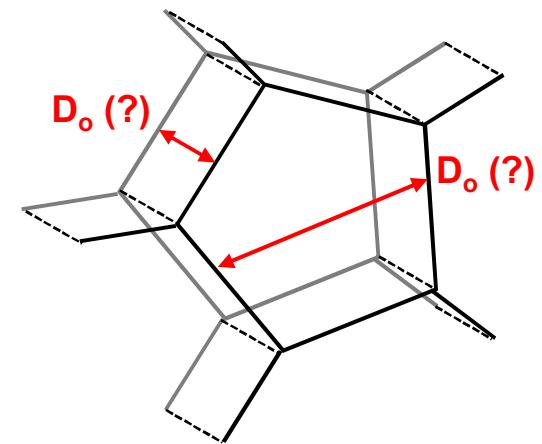
- ◆ The critical strain value, ϵ_c , is calculated using the following expression:

$$\epsilon_c = A D_o^m Z^n$$

where:

- A, m, and n are constants
- D_o , initial grain size*
- Z, Zener-Hollomon parameter

$$Z = d\epsilon/dt [\exp(\Delta H/RT)]$$



Take into account the three dimensional geometry of grains due to the thin film nature of the problem.

** D_o represents, actually, the volume of material in which the deformation must “build up” to achieve ϵ_c .*

Introduction

- ◆ The controlling equations for DRX are:

$$\varepsilon_c = A D_o^m Z^n$$

$$Z = d\varepsilon/dt [\exp(\Delta H/RT)]$$

- ◆ The constants, m and n , are both greater than zero ($m, n > 0$).
- ◆ The likelihood for DRX is maximized, when ε_c is minimized:



- Minimize the initial grain size, D_o ,

- Minimize Z , by:



- Slow strain rate, $d\varepsilon/dt$,

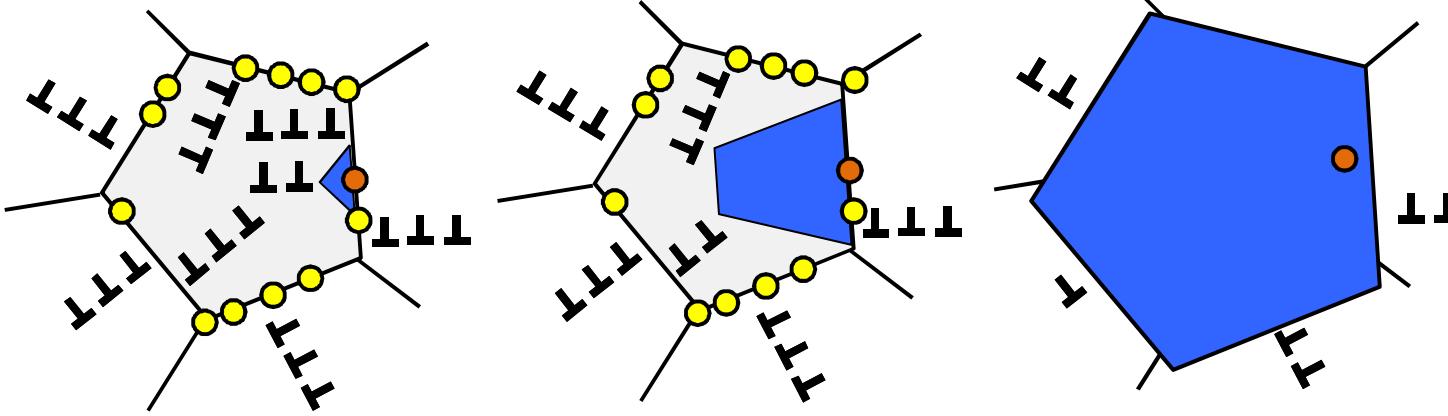
- Elevated temperatures, T

- Low apparent activation energy, ΔH

Introduction

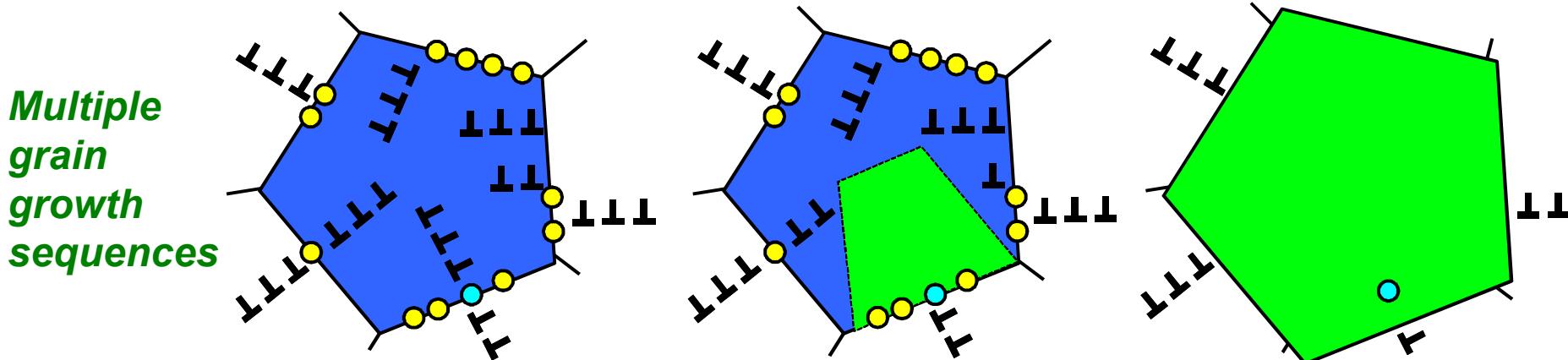
- ◆ There are two variants of dynamic recrystallization (DRX):

Continuous DRX



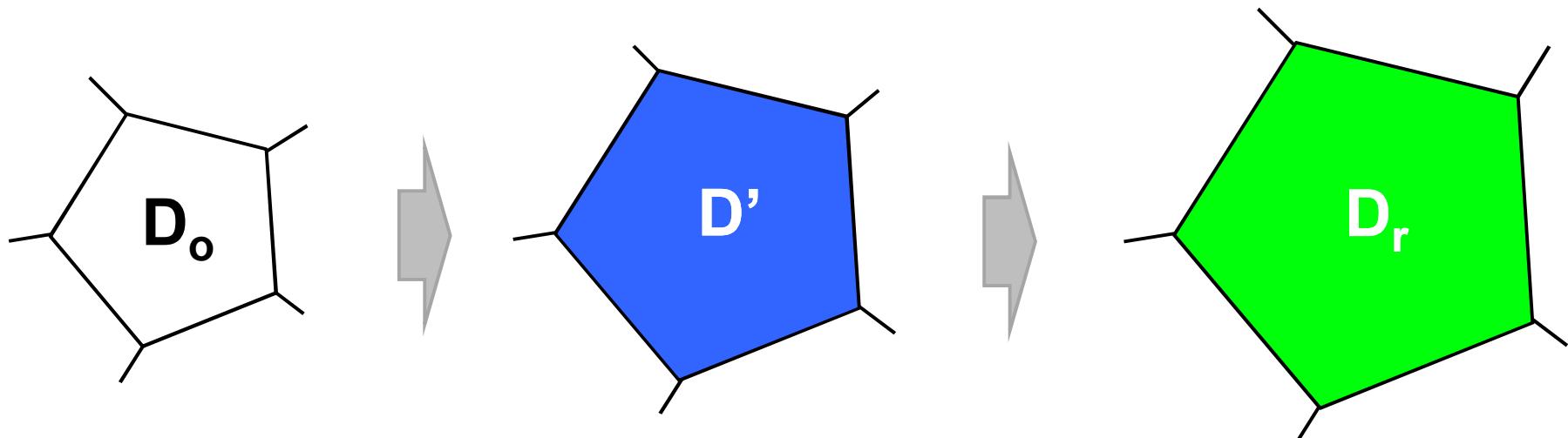
*The
End*

Cyclic DRX



Introduction

- ◆ Long whisker and hillock growth require cyclic DRX.
- ◆ The difference between the **initial grain size, D_o** , and the **final grain size, D_r** , determines the extent of DRX and as such, whether DRX is cyclic or continuous (single cycle).



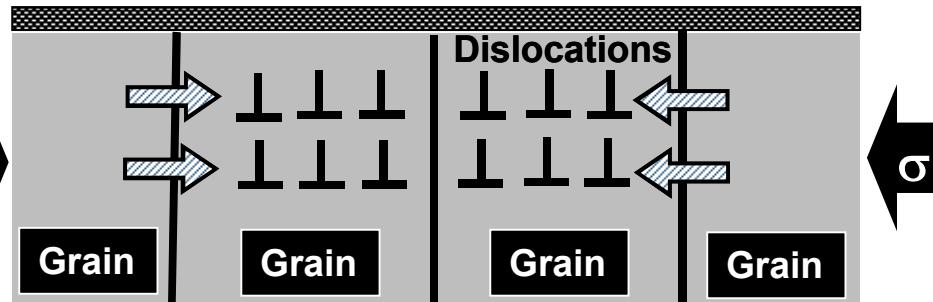
- ◆ Cyclic DRX is most likely to take place when:

$$D_o < 2D_r$$

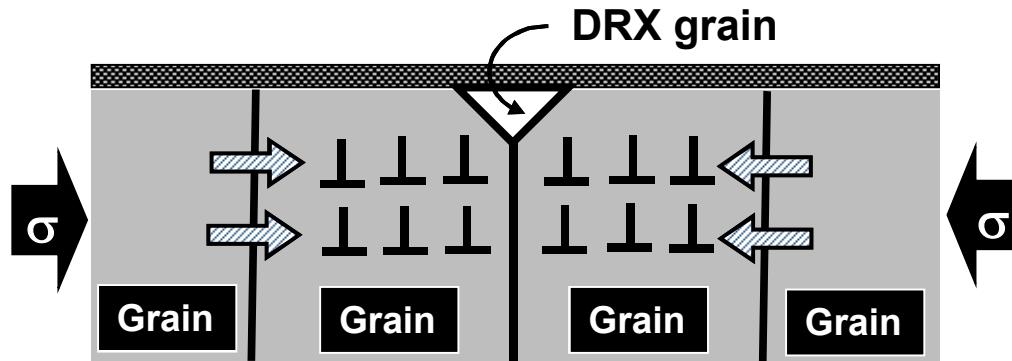
Introduction

- ◆ Diagrams show DRX scenarios vis-à-vis long whisker formation.

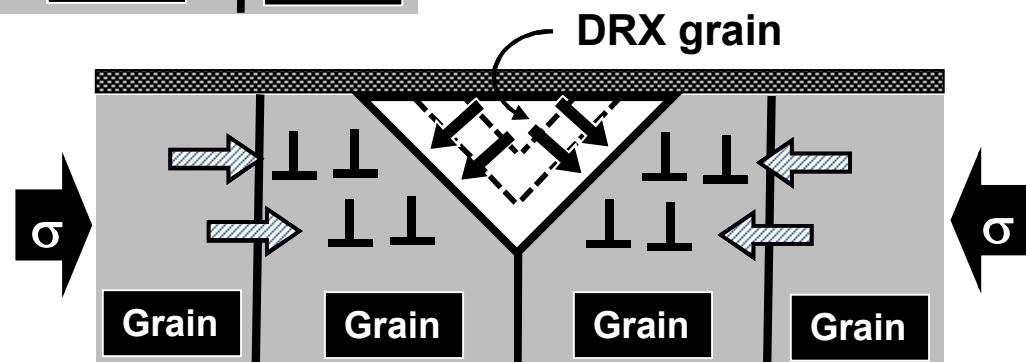
Surface oxide



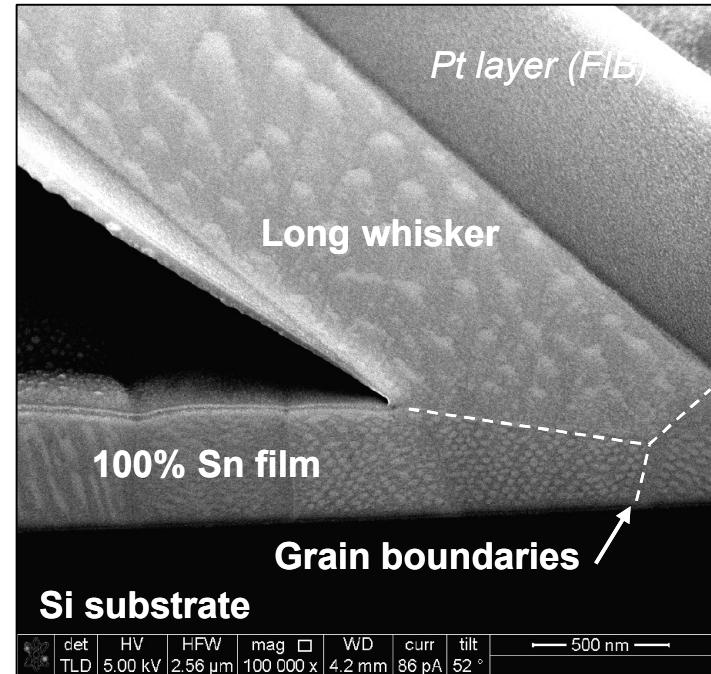
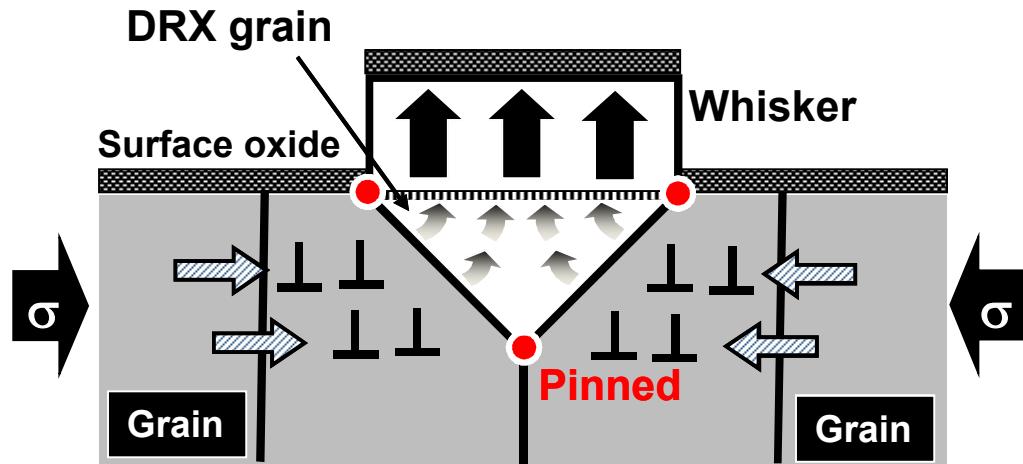
DRX grain



DRX grain



Introduction

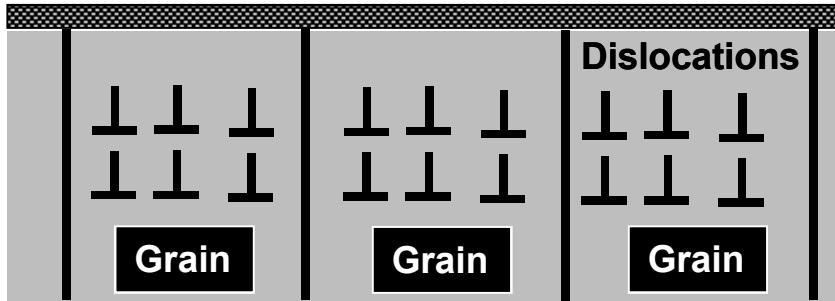


- ◆ The long whisker appears at a pre-existing grain boundary.
- ◆ The focused ion beam (FIB) cross section shows the relationship between the whisker thin film microstructures.

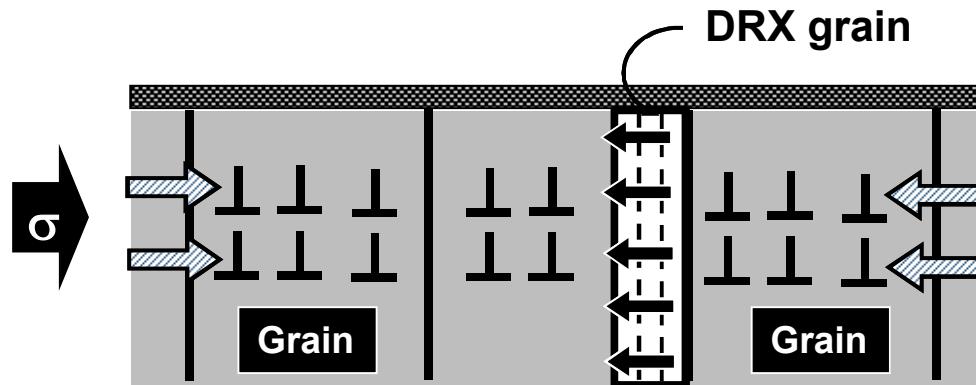
Introduction

- ◆ Here is another configuration ...

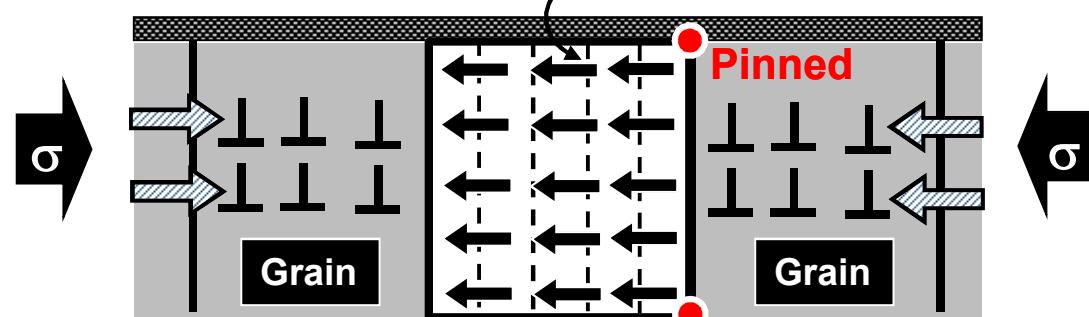
Surface oxide



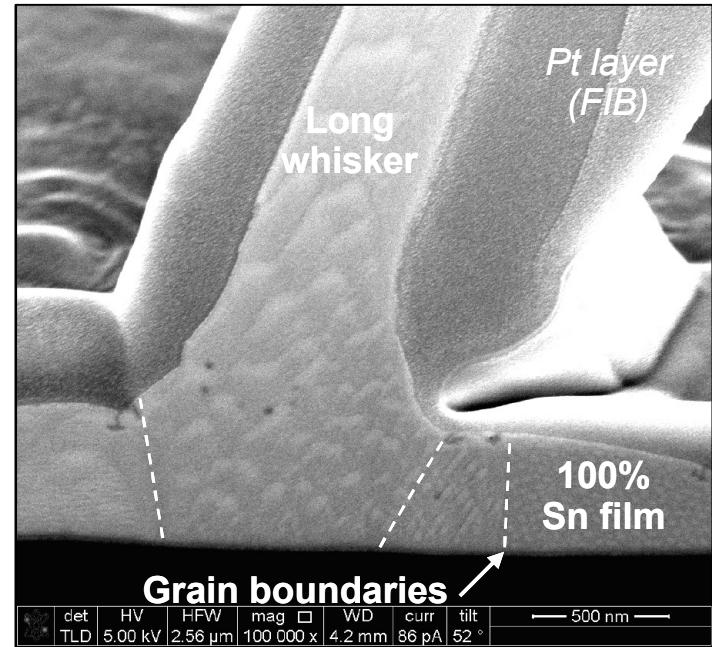
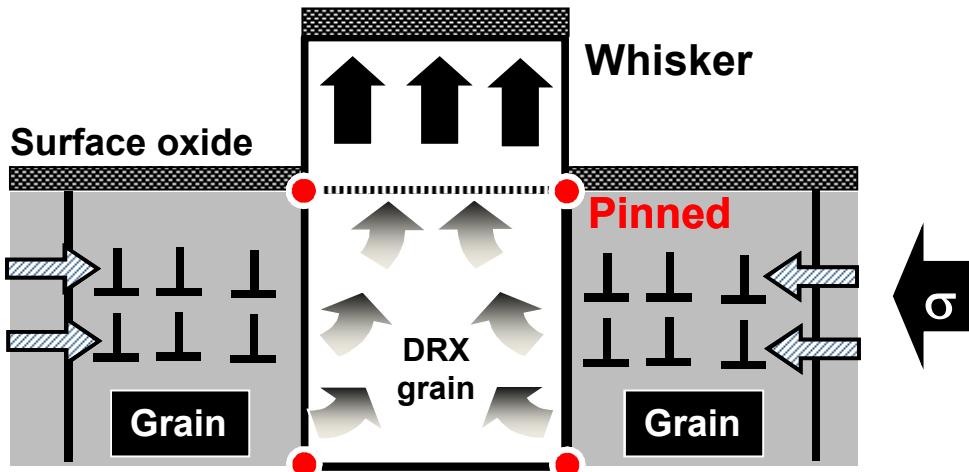
DRX grain



DRX grain



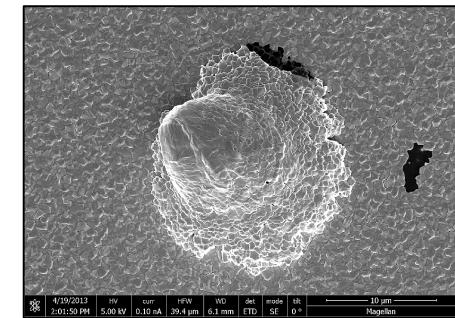
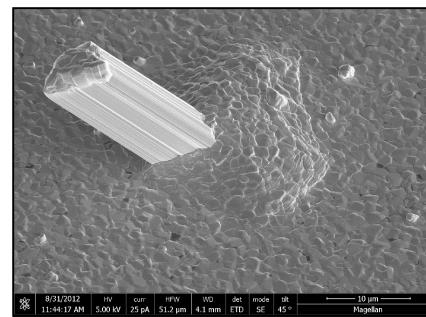
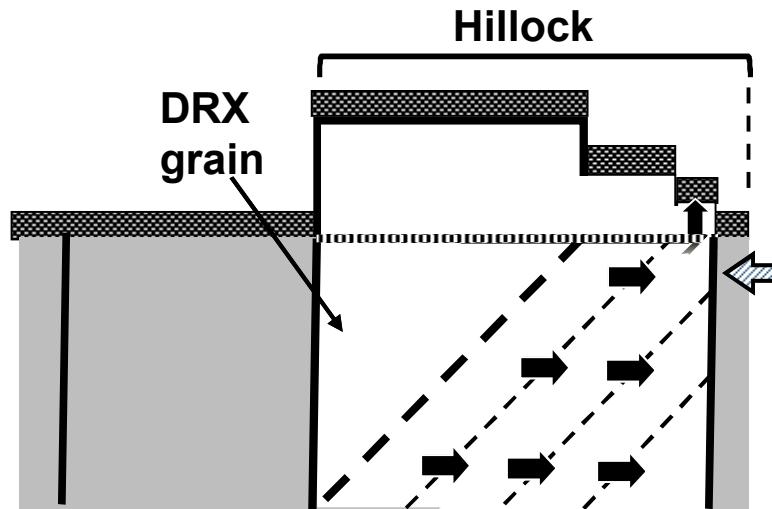
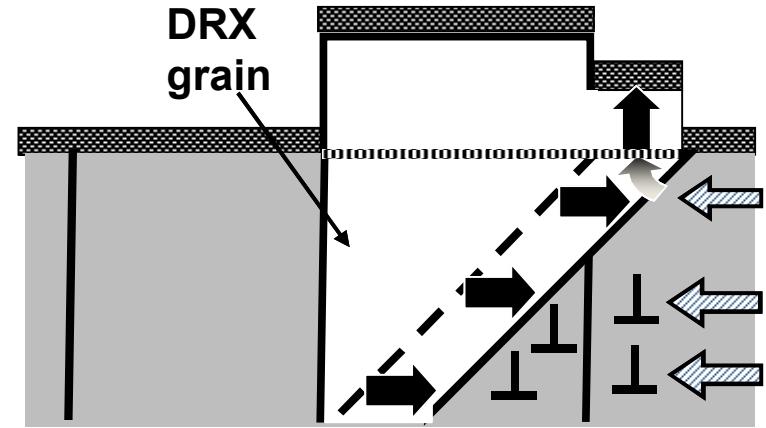
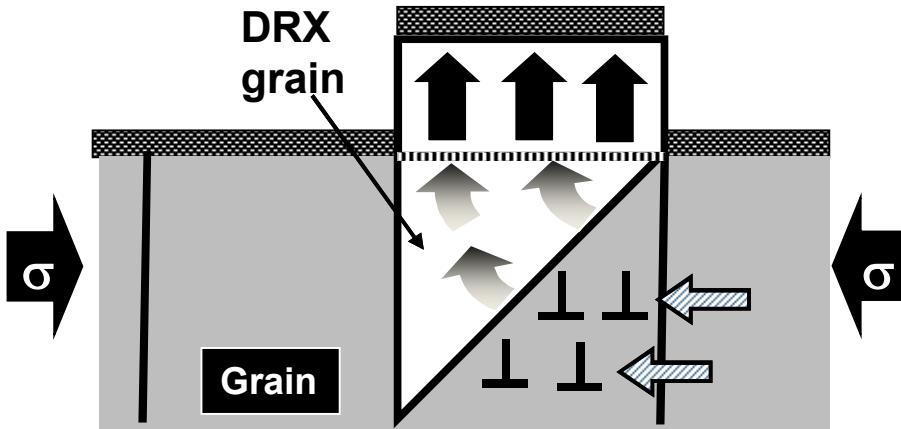
Introduction



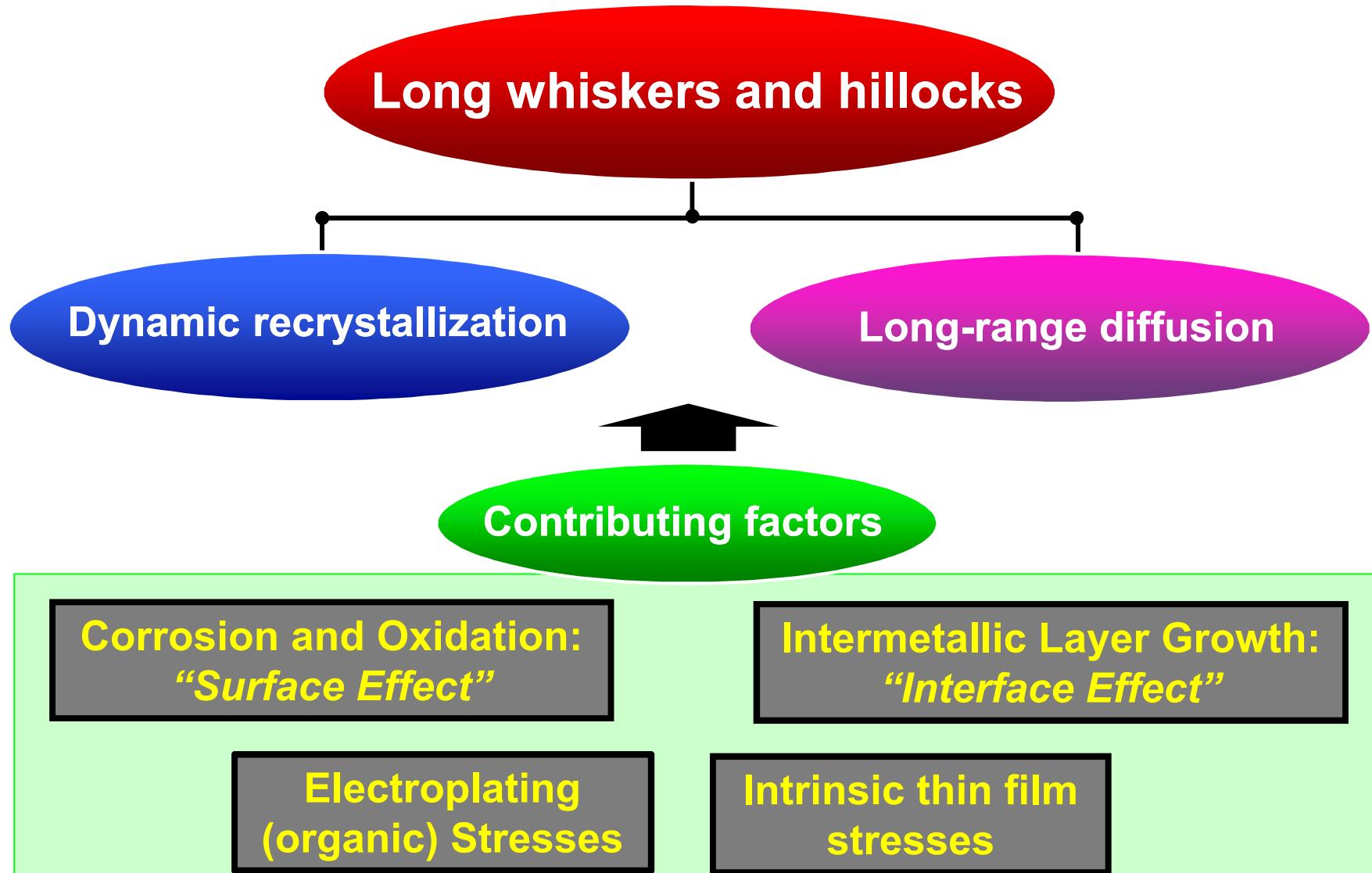
- ◆ The long whisker appears to grow from the film/substrate (20 nm Cr) interface.
- ◆ There are as many long whisker “morphologies” as there are initiation sites for DRX within the thin film microstructure.

Introduction

- ◆ Hillocks form by DRX when *grain boundary pinning is absent*.

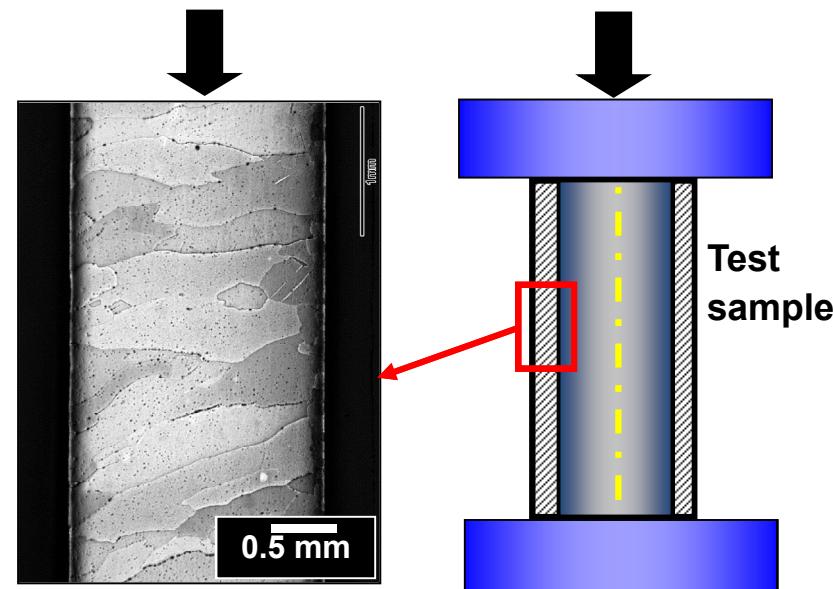


Introduction



Creep Data of Sn

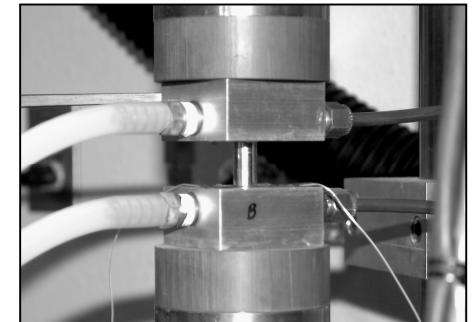
- ◆ The premise was made early on that **creep relaxation provided the driving force (strain energy) for the DRX**.
- ◆ The behavior of long whiskers and hillocks, including findings from the present study, indicate that **long-range diffusion is not the controlling factor**.
- ◆ An understanding of creep rate kinetics provides an avenue on the way to defining the kinetics of whisker formation.
- ◆ The creep experiments used **stress, temperature, time, and thermal annealing** parameters that simulated the conditions prevalent during **actual whisker growth**.



Creep Data of Sn

◆ Test conditions:

- Temperatures ... 0, 25, 50, 75, and 100°C
- Stresses ... 1.0, 2.0, 5.0, and 10.0 MPa
- Sample conditions ...
 - As-fabricated,
 - Annealed: 150°C, 24 hours or 200°C, 24 hours



Compression testing

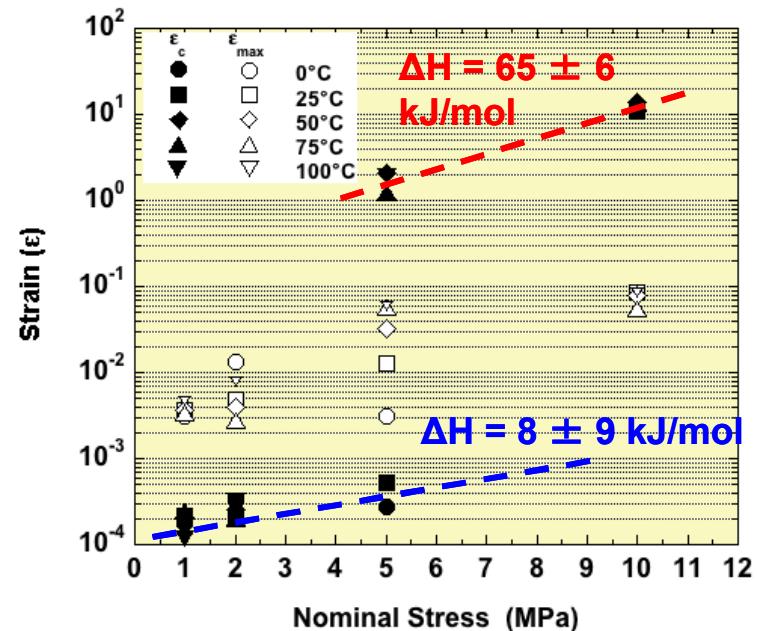
◆ Critical finding: There are two creep deformation regimes in Sn that are determined by the strain rate and identified by different ΔH values:

- $d\varepsilon/dt < 10^{-7} \text{ s}^{-1}$...

$$\Delta H = 8 \pm 9 \text{ kJ/mol}$$

- $d\varepsilon/dt > 10^{-7} \text{ s}^{-1}$...

$$\Delta H = 65 \pm 6 \text{ kJ/mol}$$

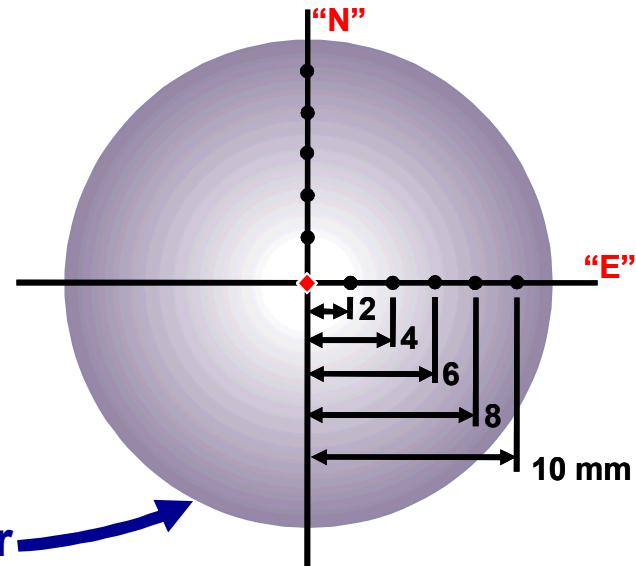
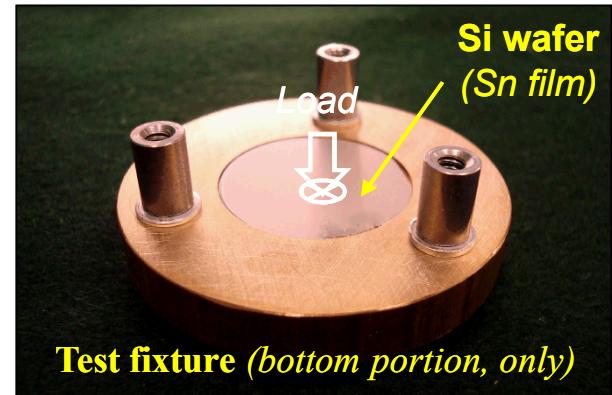


Validation of the DRX-based Model

- ◆ The evaporated Sn-on-Si test sample was developed in order to remove those “contributing factors” from the test results.

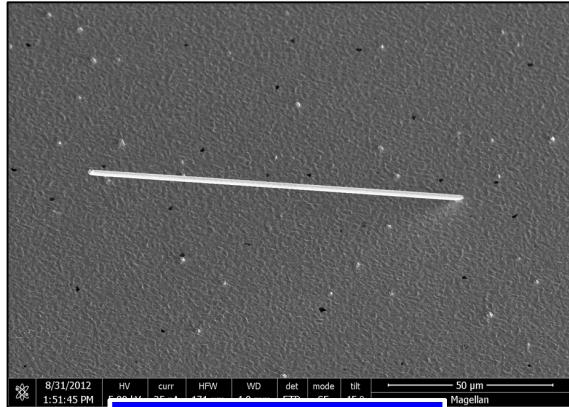
- ◆ Test parameters:

- Si wafers ... 2.54 mm diameter
... 0.275 mm thick
- Adhesion layer ... 20 nm Cr
- Sn thicknesses ...
0.25, 0.50, 1.0, 2.0, and 4.9 μm
- Temperatures ...
35, 60, 100, 120, and 150°C
- Time period ... nine (9) days
- Load ... 500g (C), 500 (T), and No Load
- Map of observation locations on the wafer

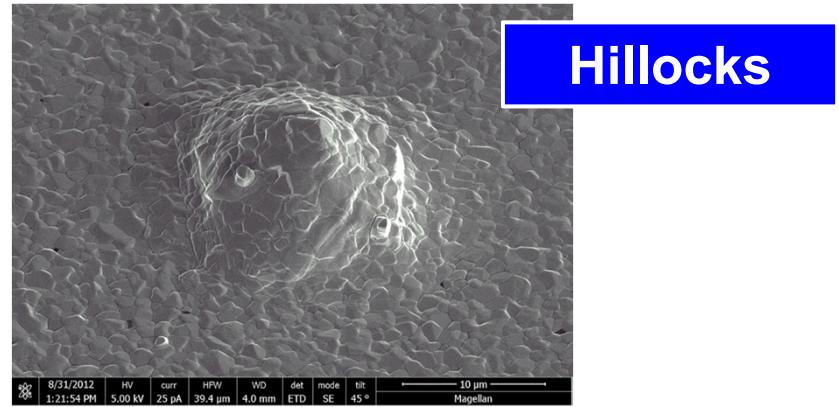


Validation of the DRX-based Model

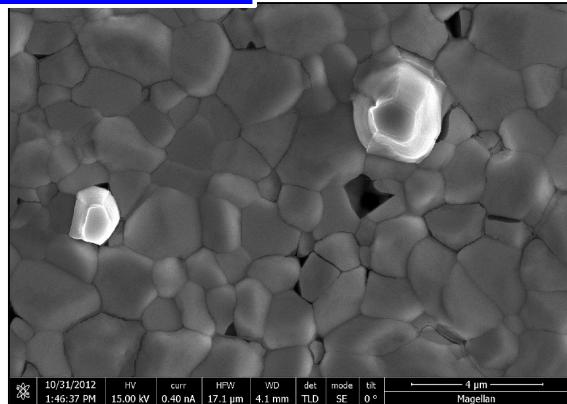
- ◆ There were three phenomenon observed in these experiments:



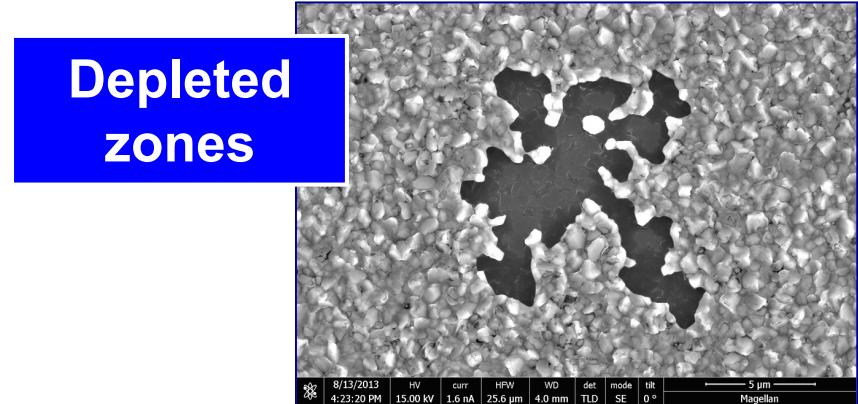
Long



Hillocks



Short,
stubby



Depleted
zones

- ◆ Required measurement granularity:

Present

or

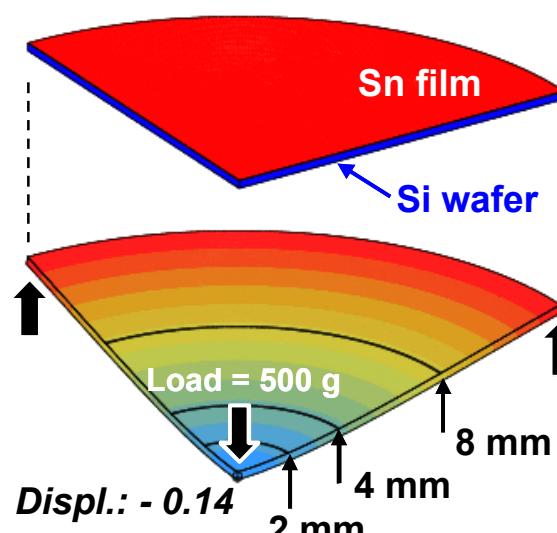
Not-present

Validation of the DRX-based Model

- ◆ Computational modeling was used to predict the anelastic strains and strain rates in the Sn films.
- ◆ Sources of anelastic strain are:
 - Applied load
 - Mismatch of coefficient of thermal expansion (CTE) between Sn and Si
- ◆ The constitutive behavior was based on the unified creep plasticity (UCP) equation for 95.5Sn-3.9Ag-0.6Cu (wt.%) solder.

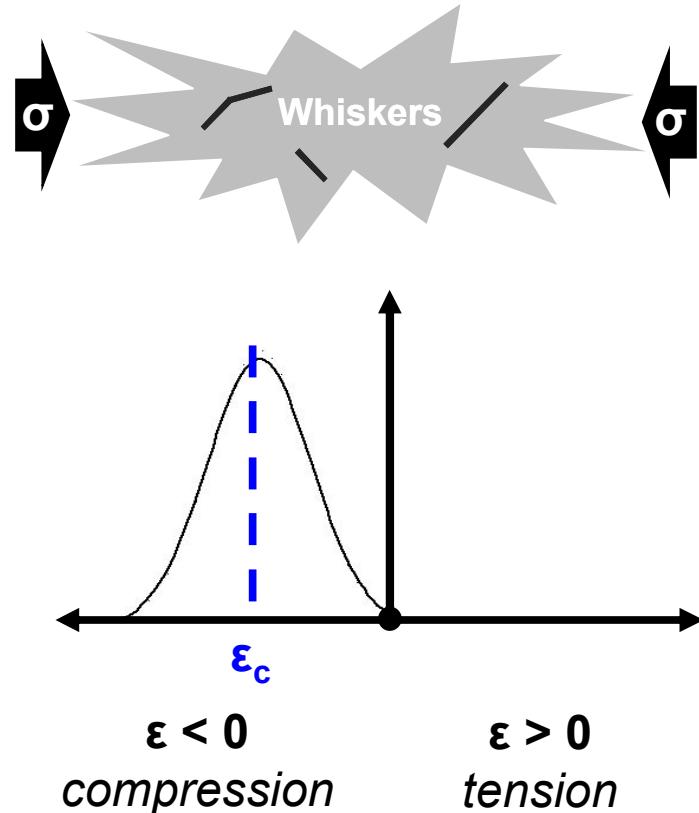
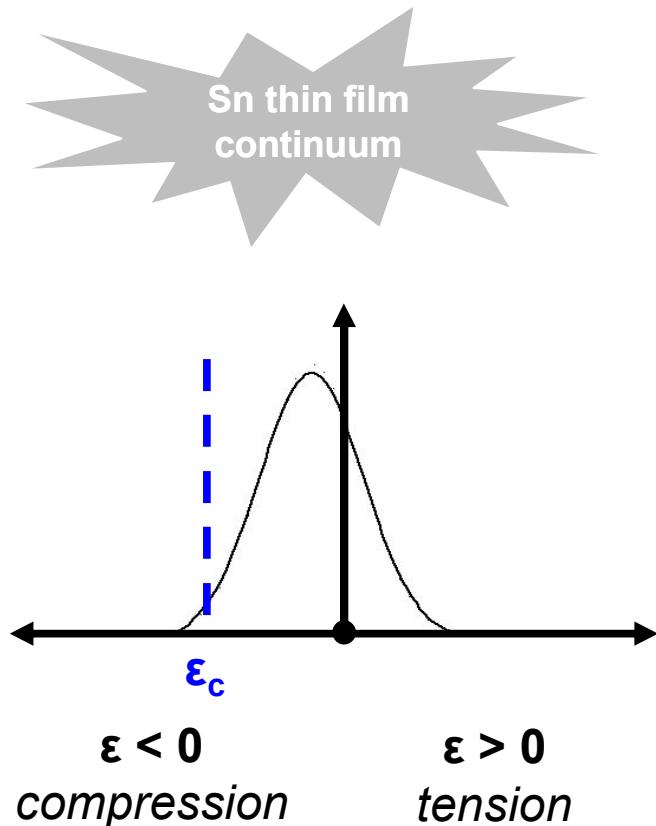
Assumptions ...

- Sn-Ag-Cu was an adequate surrogate for 100Sn.
- Von Mises strain metric – equivalent plastic strain (EQPS.)
- *Continuum mechanics model is applied to a single grain behavior.*
(con't)



Validation of the DRX-based Model

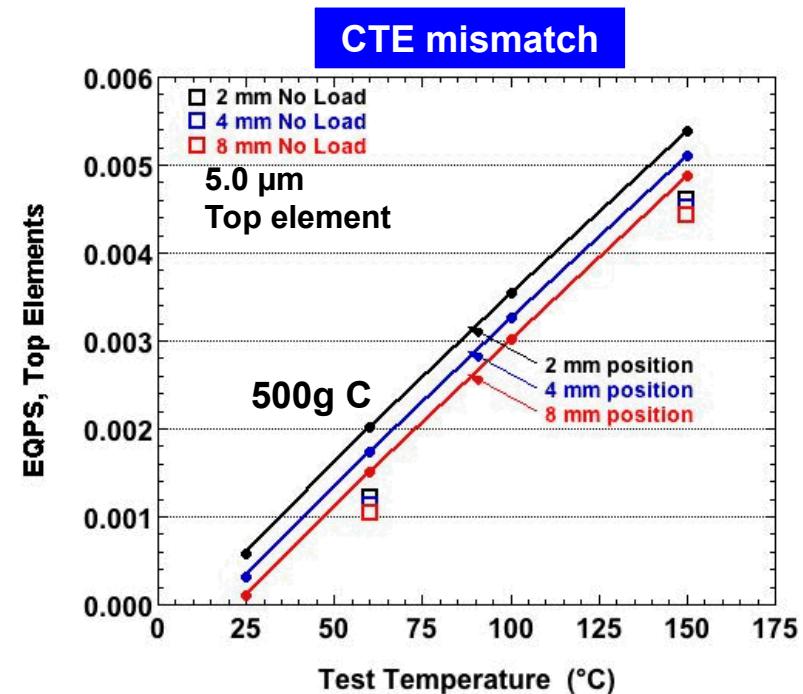
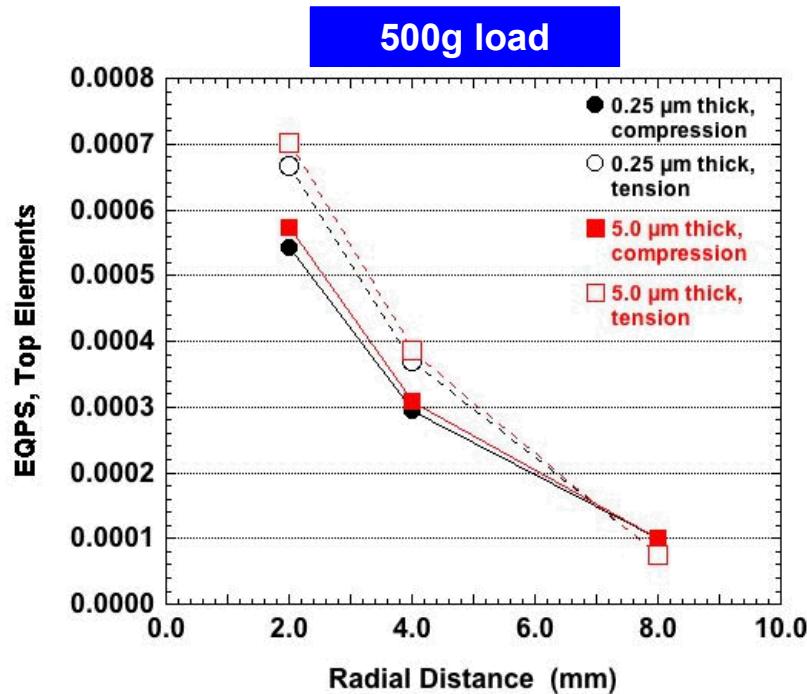
- ◆ Continuum mechanics assumption warrants further explanation:



- ◆ The **global viewpoint** is taken that whiskers, hillocks, and depleted zones form from a thin film continuum comprised of the individual Sn grains.

Validation of the DRX-based Model

- ◆ The computational model identified the following insights:



- EQPS: $10^{-4} - 10^{-3}$.
- $EQPS_{0.25\ \mu\text{m}} \approx EQPS_{5.0\ \mu\text{m}}$
- Compression differs from tension, more so towards the center
- EQPS: $10^{-3} - 10^{-2}$.
- Same EQPS_{CTE} at all radial positions.
- Same EQPS_{CTE} at all film thicknesses. Because ... Si thickness $>>$ Sn thickness.

Validation of the DRX-based Model

◆ These generalizations were developed from the model predictions:

- There was a negligible strain gradient in the through-thickness dimension of each film.
- The EQPS and strain rates are similar between the 0.25 μm and 4.9 μm thicknesses.

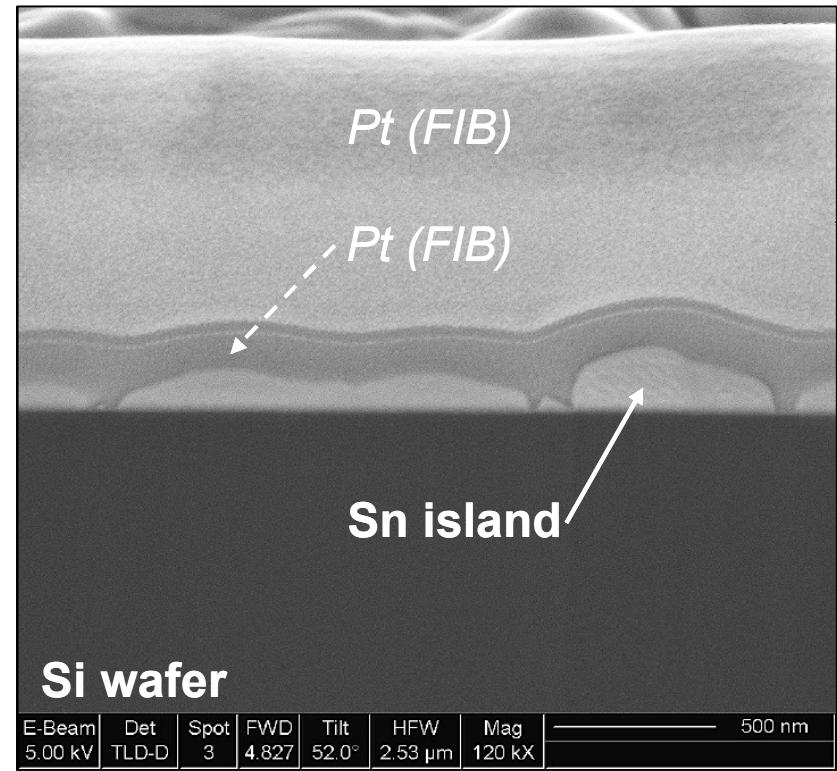
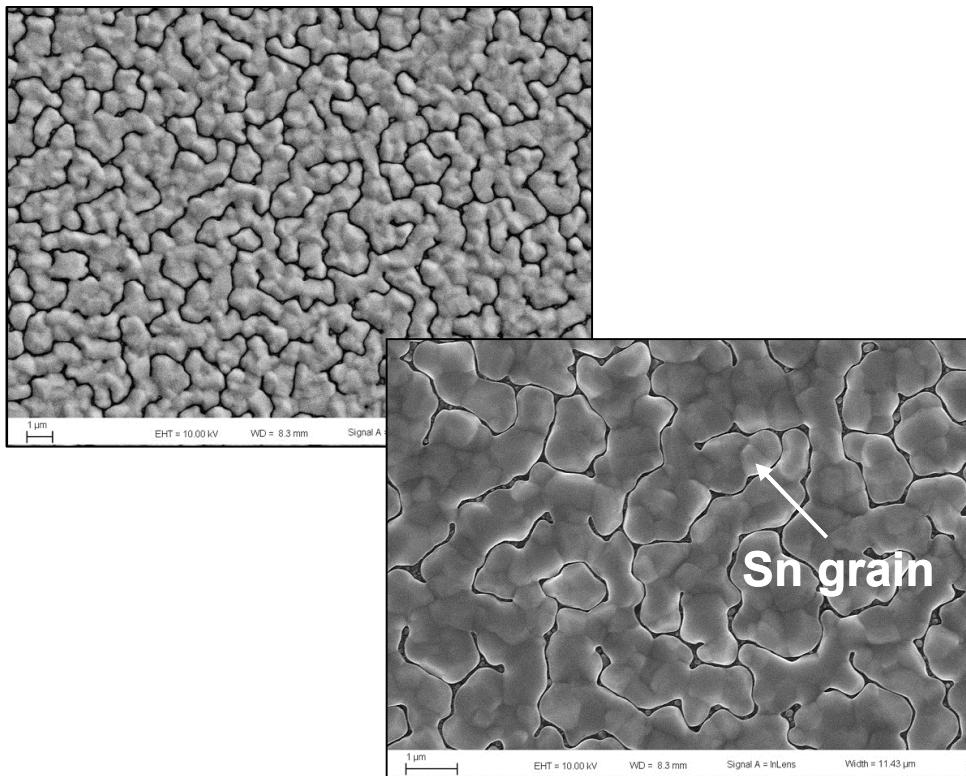
The reason is ... Si thickness >> Sn thickness.

- Therefore, it is the microstructural aspect of grain size that controls, explicitly, DRX in the Sn films.
 - Grain size, D_o , is determined primarily by film thickness.

- The CTE mismatch generates an EQPS that is an order-of-magnitude greater than those created by a 500g load.
 - The mechanical load provides a “fine-tuning” to the EQPS values.

Validation of the DRX-based Model

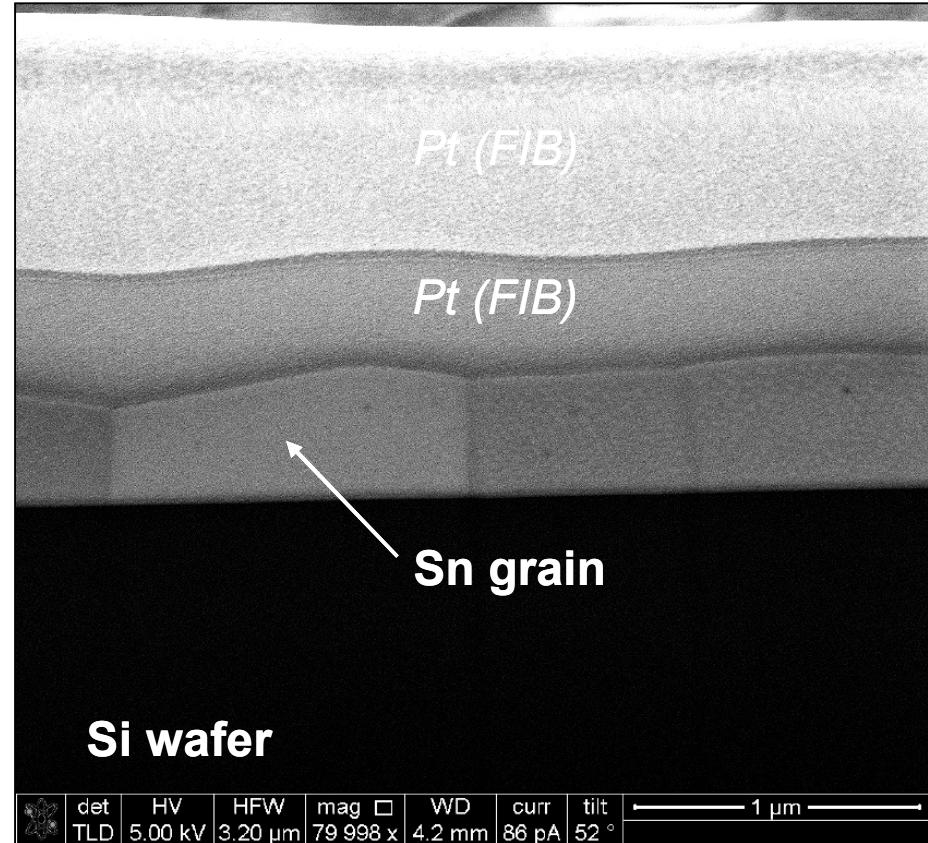
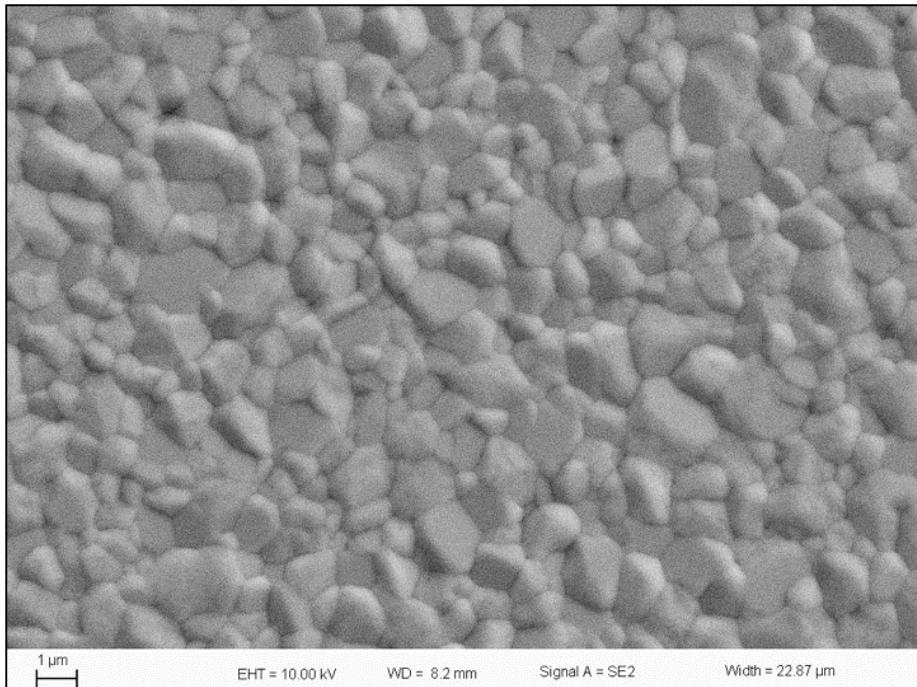
- ◆ The as-fabricated, 0.25 μm Sn film exhibited an “island” structure.
- ◆ The “islands” were interconnected over extended distances.



0.25 μm

Validation of the DRX-based Model

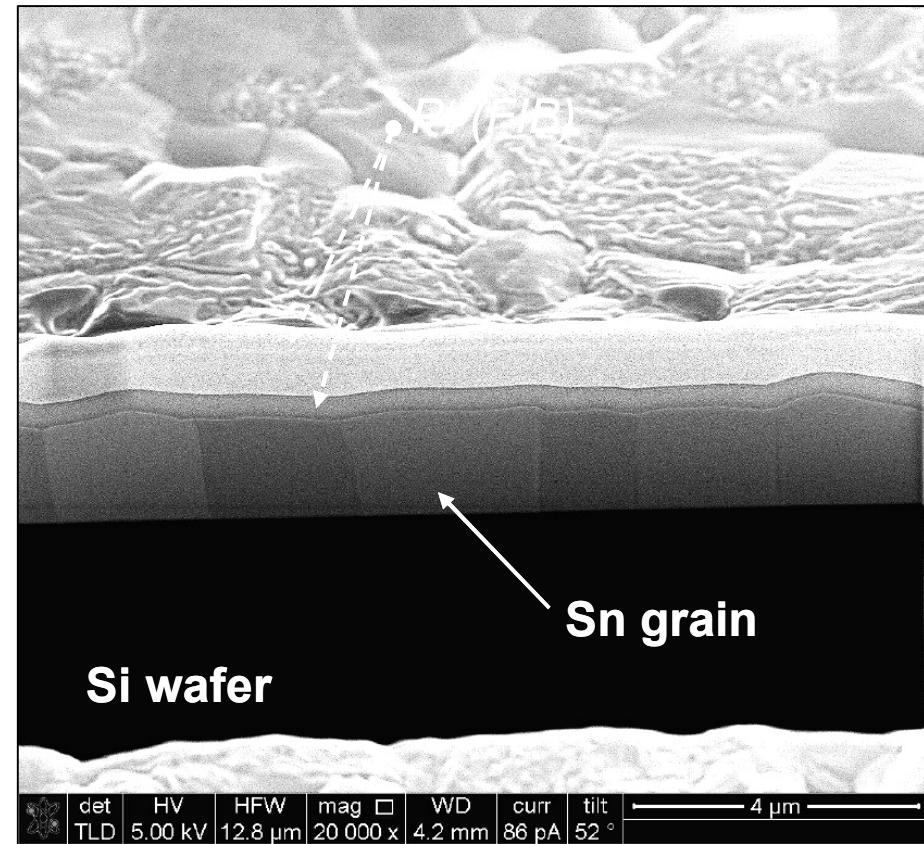
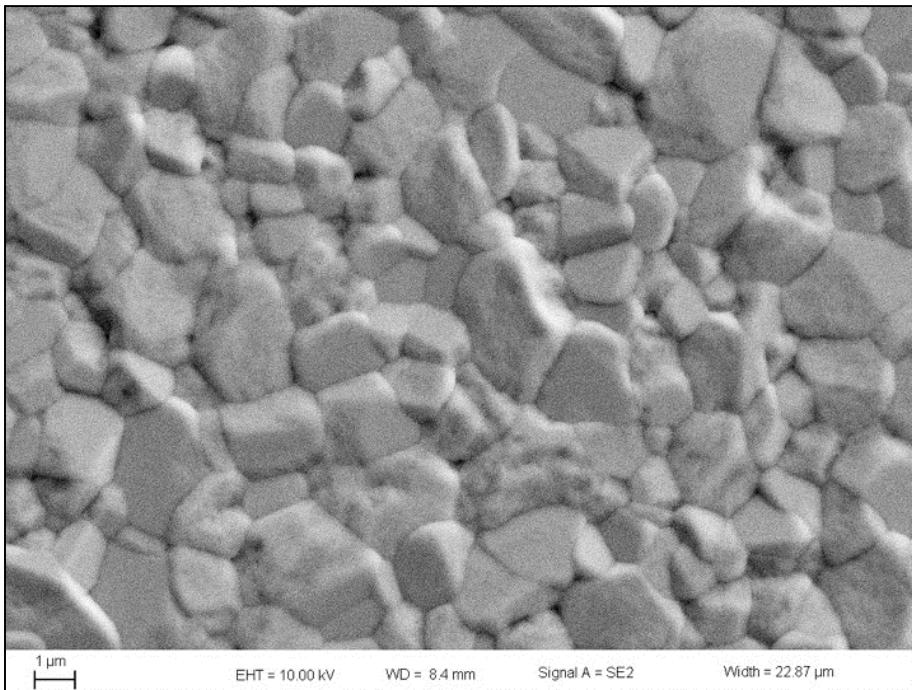
- ◆ The 0.5 μm and 1.0 μm thin films exhibited a columnar structure.



0.50 μm

Validation of the DRX-based Model

- ◆ The 2.0 μm and 4.0 μm thin films also exhibited columnar microstructures in the thickness direction.



2.0 μm

Validation of the DRX-based Model

◆ The following generalizations pertained to the Sn films:

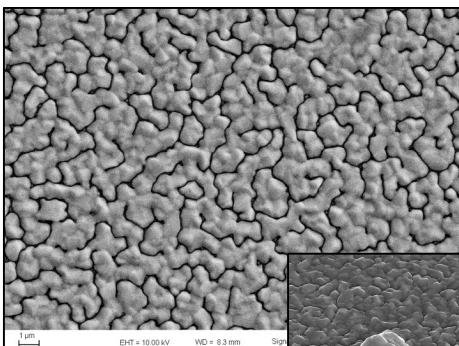
- The microstructures were columnar through the thickness.
- The in-plane grain sizes did not vary significantly between the different film thickness:
 - 0.25 μm < 1 μm
 - 0.5 μm < 1 μm to 3 – 4 μm
 - 1.0 μm < 1 μm to 3 – 4 μm
 - 2.0 μm < 1 μm to 3 – 4 μm
 - 4.9 μm 1 μm to 5 μm
- Aside from the 0.25 μm “island” structure; thickness differences; and slight variations of in-plane grain size, there were no other artifacts that predisposed the films to different behaviors.

Validation of the DRX-based Model

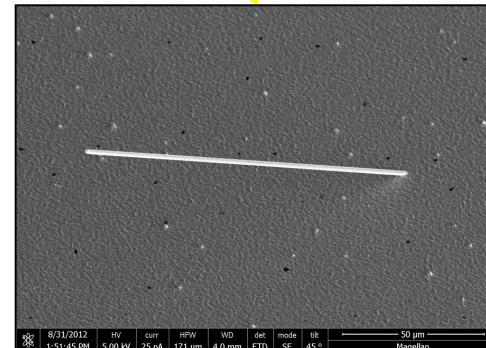
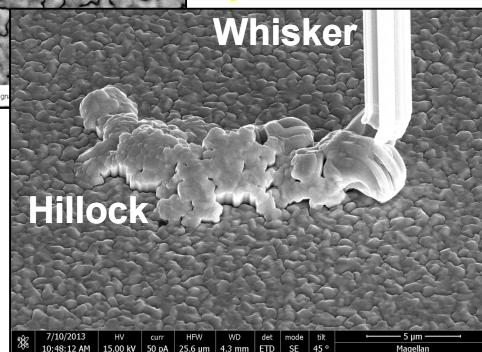
- ◆ The table describes the observation of long-whiskers:

Film Thickness	Temperature (C)				
	35	60	100	120	150
0.25	C T N	C T N	C T N	C T N	C T N
0.5	C T N	C T N	C T N	C T N	C T N
1.0	C T N	C T N	C T N	C T N	C T N
2.0	C T N	C T N	C T N	C T N	C T N
4.9	C T N	C T N	C T N	C T N	C T N

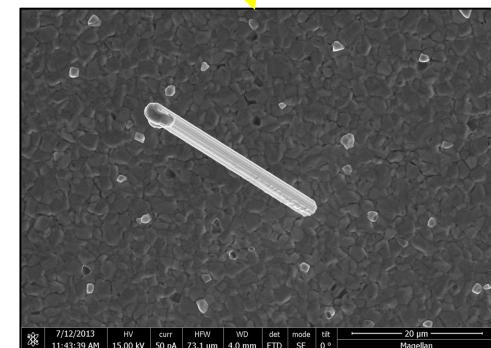
Green is present; red is absent. "C," compression; "T," tension; and "N," no-load



0.25 μm



(0.5 μm) 1.0 μm



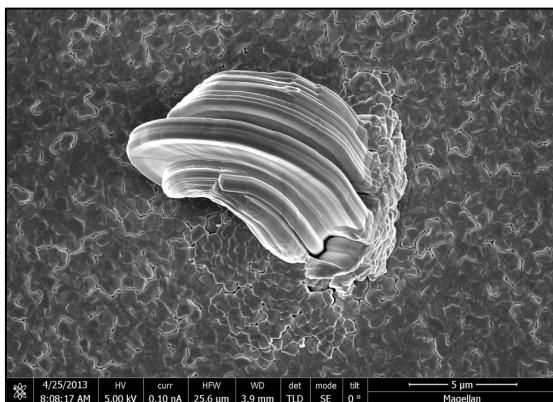
2.0 μm (4.9 μm)

Validation of the DRX-based Model

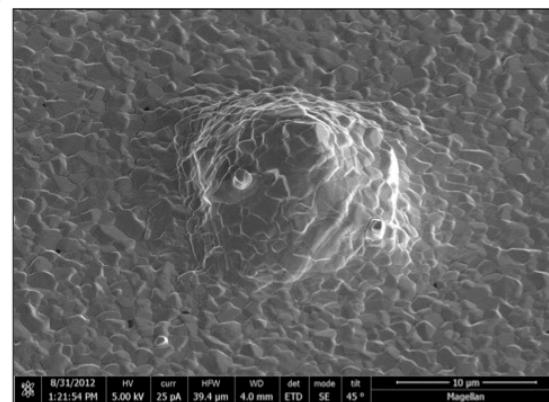
- ◆ The table describes the observation of **hillocks**:

Film Thickness	Temperature (C)				
	35	60	100	120	150
0.25	C T N	C T N	C T N	C T N	C T N
0.5	C T N	C T N	C T N	C T N	C T N
1.0	C T N	C T N	C T N	C T N	C N
2.0	C T N	C T N	C T N	C T N	C T N
4.9	C T N	C T N	C T N	C T N	C T N

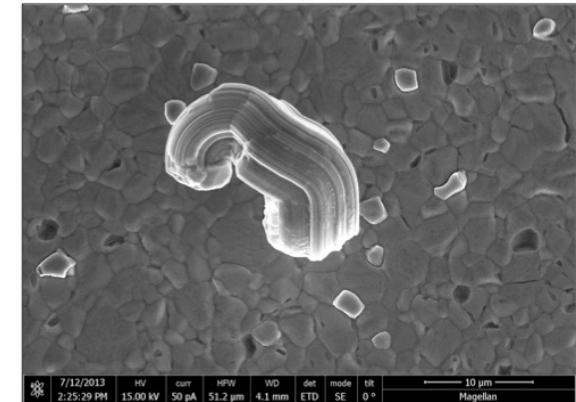
Green is present; red is absent. "C," compression; "T," tension; and "N," no-load



0.25 μm



1.0 μm



2.0 μm

- ◆ Hillocks were generally prevalent when long-whiskers were not so.

Validation of the DRX-based Model

◆ The table combines long-whiskers and hillocks to represent DRX.

Film Thickness	Temperature (C)				
	35	60	100	120	150
0.25	Red	Red	Green	Green	Green
0.5	Red	Green	Green	Green	Green
1.0	Green	Green	Green	Green	Green
2.0	Red	Red	Red	Red	Green
4.9	Red	Red	Red	Red	Green

Green is present; red is absent.

The white boundary shows the interior region (green) where DRX is prevalent.

◆ These general observations were established from the data:

- There was DRX activity in the 0.25 μm films.
 - Hillocks, primarily \rightarrow Absence of grain boundary pinning
- DRX activity as most prevalent in the 0.5 μm and 1.0 μm films.
 - 0.5 μm : whiskers, only \rightarrow Absence of grain boundary pinning
 - 1.0 μm : mixture of whiskers and hillocks \rightarrow Variable
- DRX activity dropped off sharply for the 2.0 μm and 4.9 μm films.
 - 2.0 μm : whiskers, only
 - 4.9 μm : hillocks, only

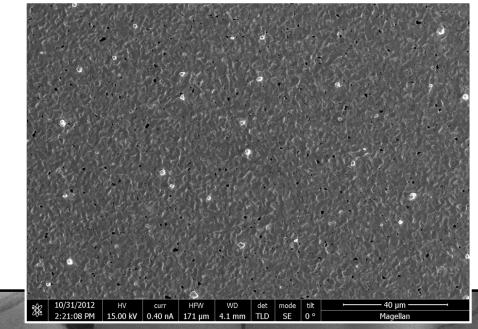
Validation of the DRX-based Model

- ◆ Long whiskers or hillocks dropped for the 2.0 μm and 4.0 μm Sn film except ...

150°C

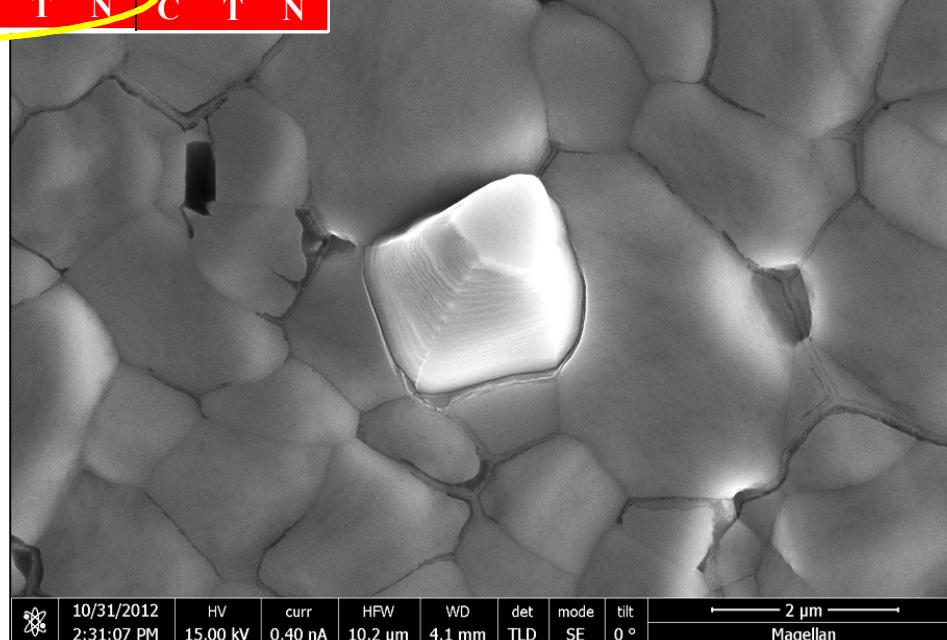
- 2.0 μm : whiskers, only
- 4.9 μm : hillocks, only

Film Thickness	Temperature (C)				
	35	60	100	120	150
0.25	C T N	C T N	C T N	C T N	C T N
0.5	C T N	C T N	C T N	C T N	C T N
1.0	C T N	C T N	C T N	C T N	C N
→ 2.0	C T N	C T N	C T N	C T N	C T N
→ 4.9	C T N	C T N	C T N	C T N	C T N



Green is present; red is absent. "C," compression; "T," tension; and "N," no-load

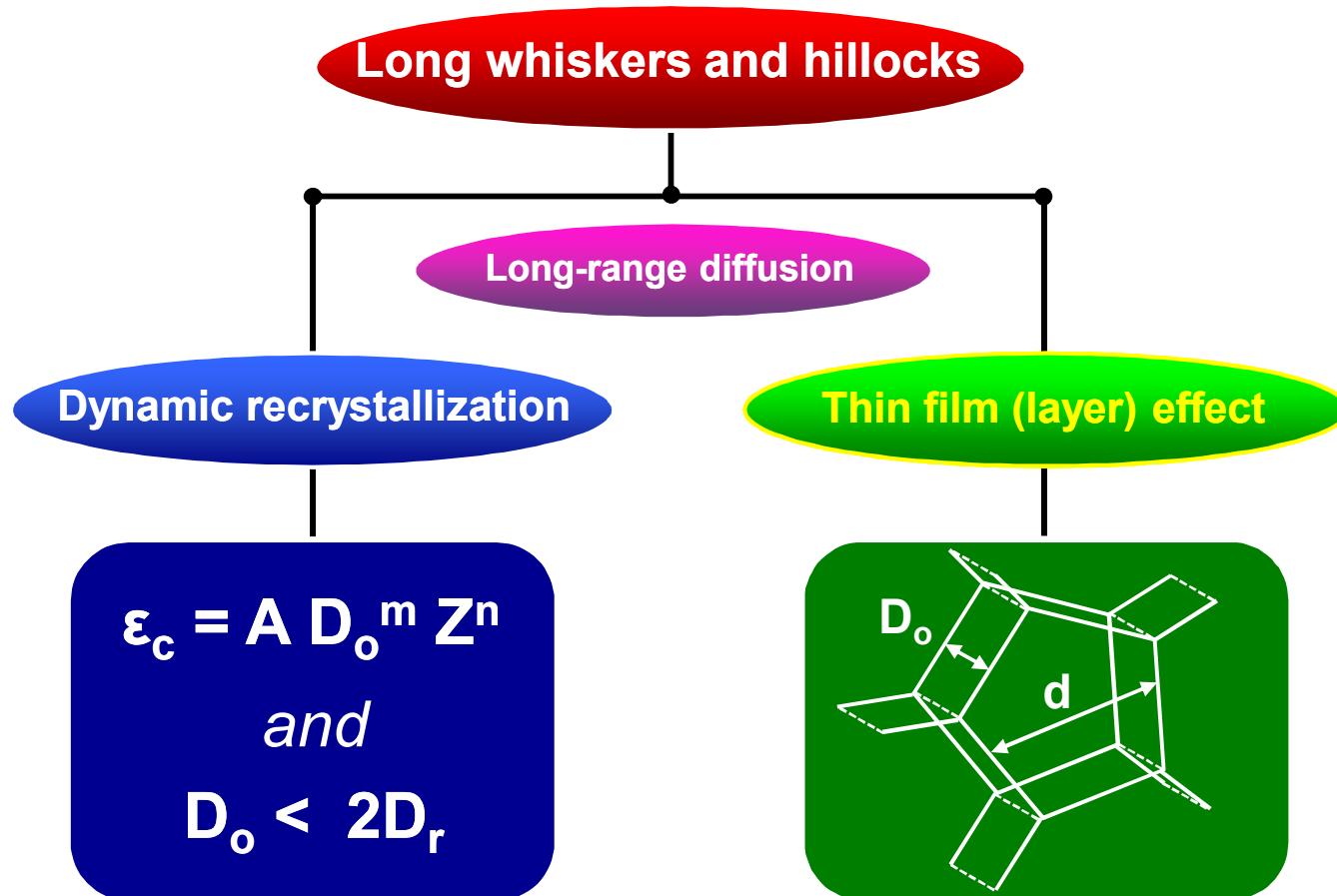
- ◆ But, short, stubby whiskers were observed on both films, lesser so on the 4.9 μm case.



The short, stubby whiskers indicated that continuous (single cycle) DRX was active.

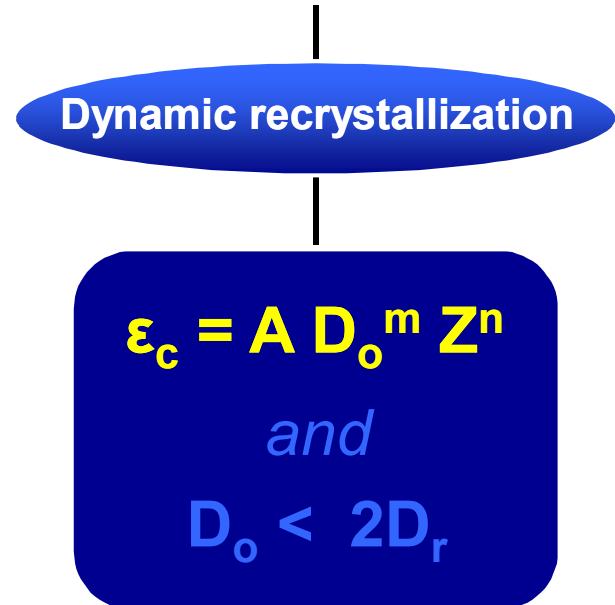
Validation of the DRX-based Model

- ◆ *What is the story ???*



Validation of the DRX-based Model

- ◆ The controlling factor is, of course, the critical strain, ε_c .
- ◆ Tin (Sn) exhibited two creep (mechanism) regimes based upon the strain rate, $d\varepsilon/dt$:
 - $d\varepsilon/dt < 10^{-7} \text{ s}^{-1}$...
 $\Delta H = 8 \pm 9 \text{ kJ/mol}$
 - $d\varepsilon/dt > 10^{-7} \text{ s}^{-1}$...
 $\Delta H = 65 \pm 6 \text{ kJ/mol}$
- ◆ The following analysis was performed:
 - Two bands of ε_c versus temperature plots were computed for all $D_o = 0.25, 0.50, 1.0, 2.0, \text{ and } 4.9 \mu\text{m}$ and a strain rate of $d\varepsilon/dt = 10^{-7} \text{ s}^{-1}$.
 - The two bands represented $\Delta H = 8 \pm 9 \text{ kJ/mol}$ or $\Delta H = 65 \pm 6 \text{ kJ/mol}$, using the A, m, and n obtained in the previous creep study.
(con't)



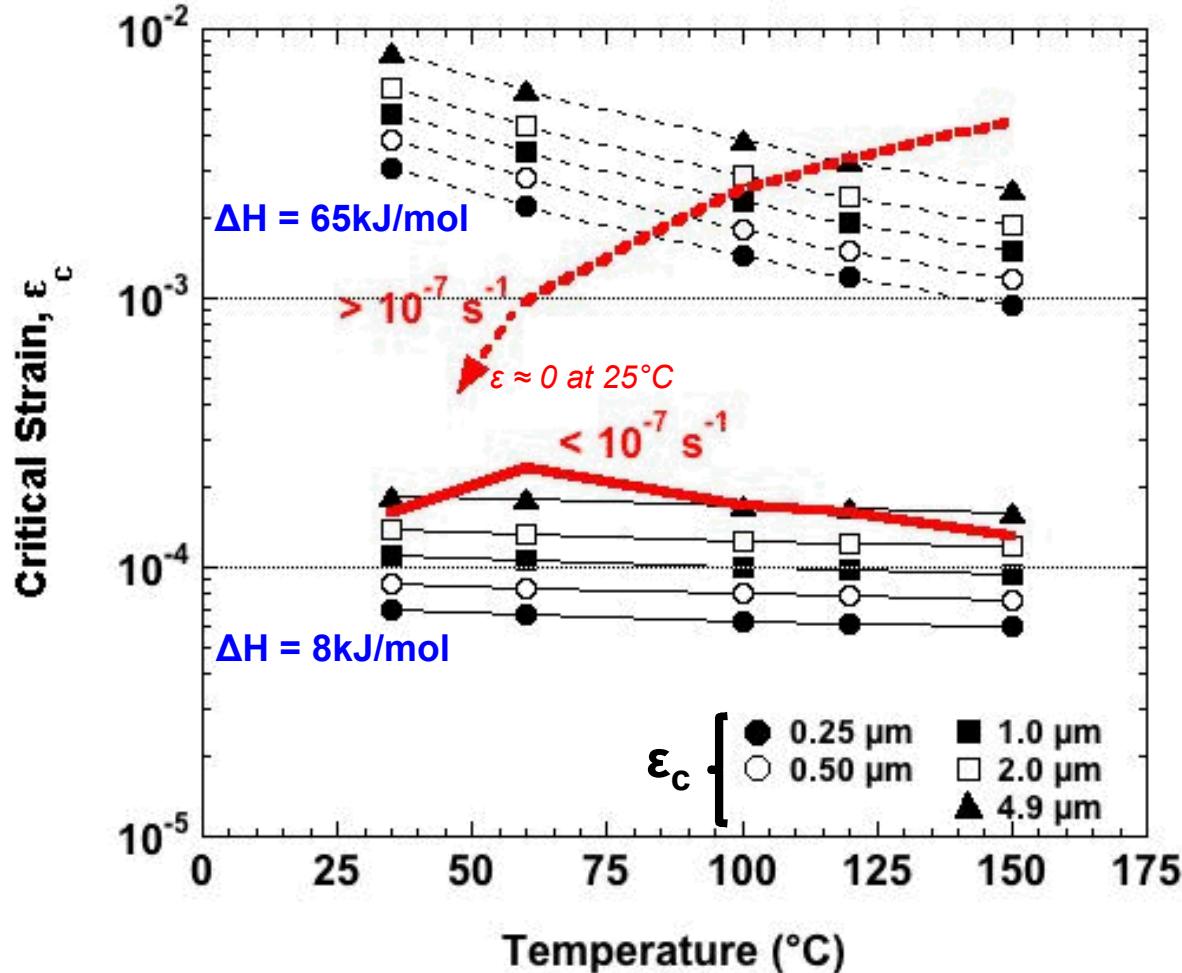
Validation of the DRX-based Model

- Secondly, the computational model was used to calculate ϵ versus temperature for all D_o thicknesses. *But ...*

The computed ϵ was partitioned into the strain accumulated under:
 $d\epsilon/dt > 10^{-7} \text{ s}^{-1}$...

... and the portion of ϵ that accumulated under:
 $d\epsilon/dt < 10^{-7} \text{ s}^{-1}$.

- It was assumed that the strain that grew in the $> 10^{-7} \text{ s}^{-1}$ regime did so independently of the strain that grew in the $< 10^{-7} \text{ s}^{-1}$ regime.

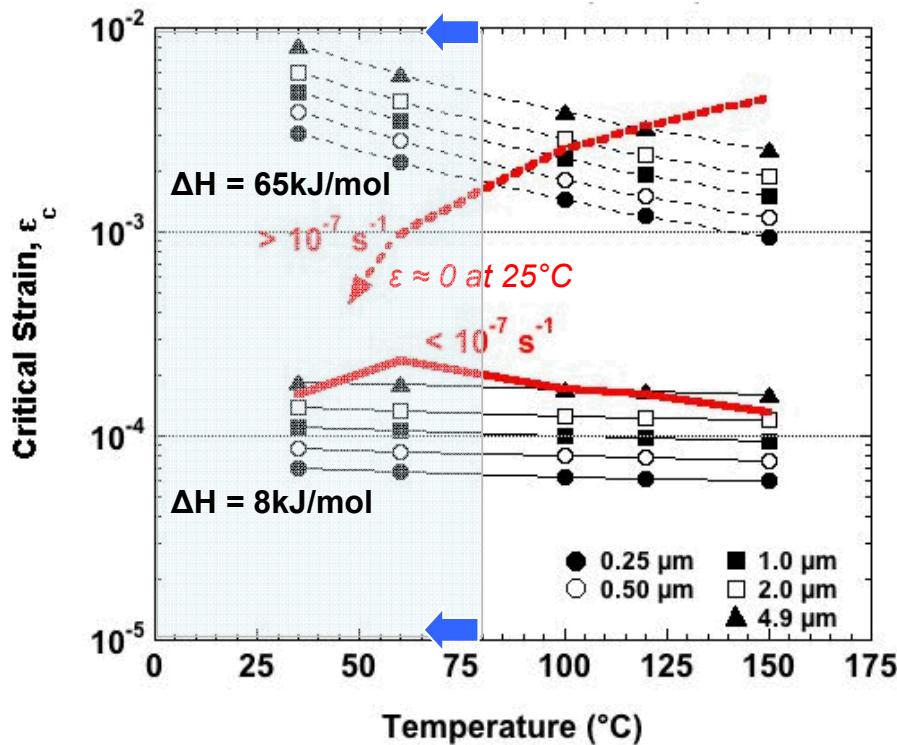


Validation of the DRX-based Model

Film Thickness	Temperature (C)				
	35	60	100	120	150
0.25	Red	Red	Green	Green	Green
0.5	Red	Green	Green	Green	Green
1.0	Green	Green	Green	Green	Green
2.0	Red	Red	Red	Red	Green
4.9	Red	Red	Red	Red	Green

- ◆ These cases will show the development of the validation ...

T < 80°C



- $d\varepsilon/dt > 10^{-7} \text{ s}^{-1}$...

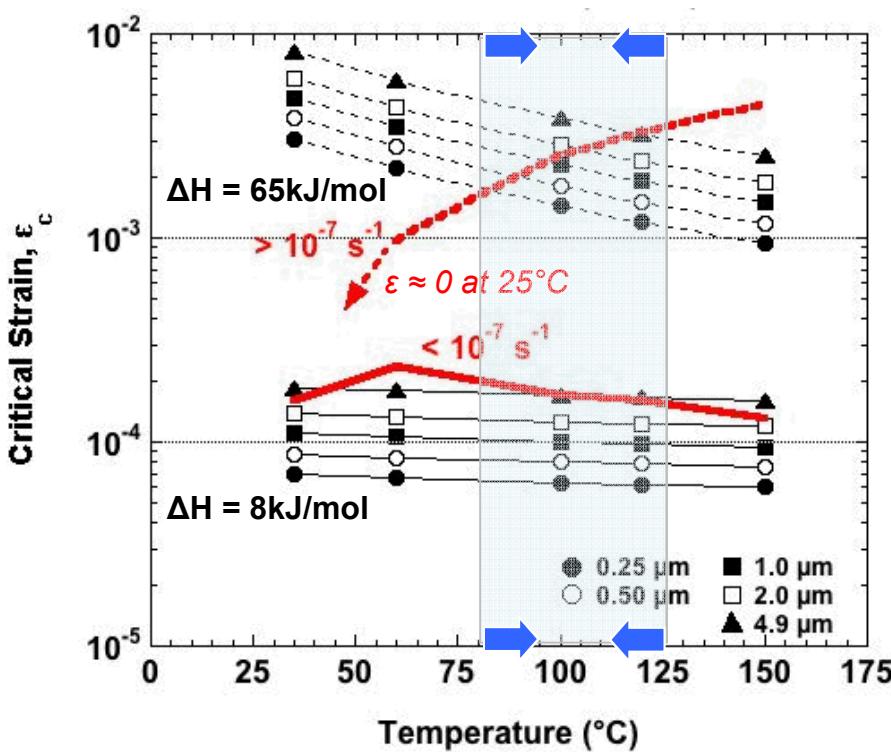
 - $\varepsilon < \varepsilon_c$, $0.25 - 4.9 \text{ mm}$
 - **No driving force for DRX.**
- $d\varepsilon/dt < 10^{-7} \text{ s}^{-1}$...

 - $\varepsilon > \varepsilon_c$, for $0.25 - 2.0 \mu\text{m}$
 - **Driving force for DRX.**
 - $\varepsilon < > \varepsilon_c$, for $4.9 \mu\text{m}$
 - **DRX ... borderline**

Validation of the DRX-based Model

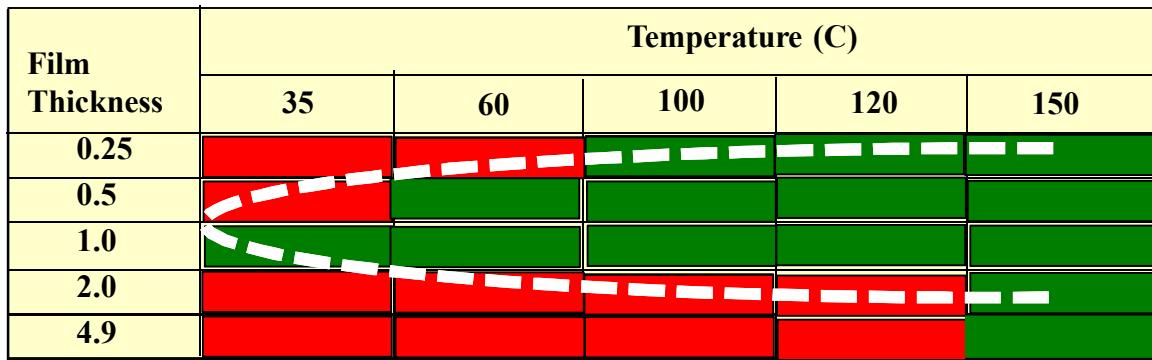
Film Thickness	Temperature (C)				
	35	60	100	120	150
0.25	Red	Red	Green	Green	Green
0.5	Red	Green	Green	Green	Green
1.0	Green	Green	Green	Green	Green
2.0	Red	Red	Red	Green	Green
4.9	Red	Red	Red	Red	Green

$80^\circ\text{C} < T < 125^\circ\text{C}$

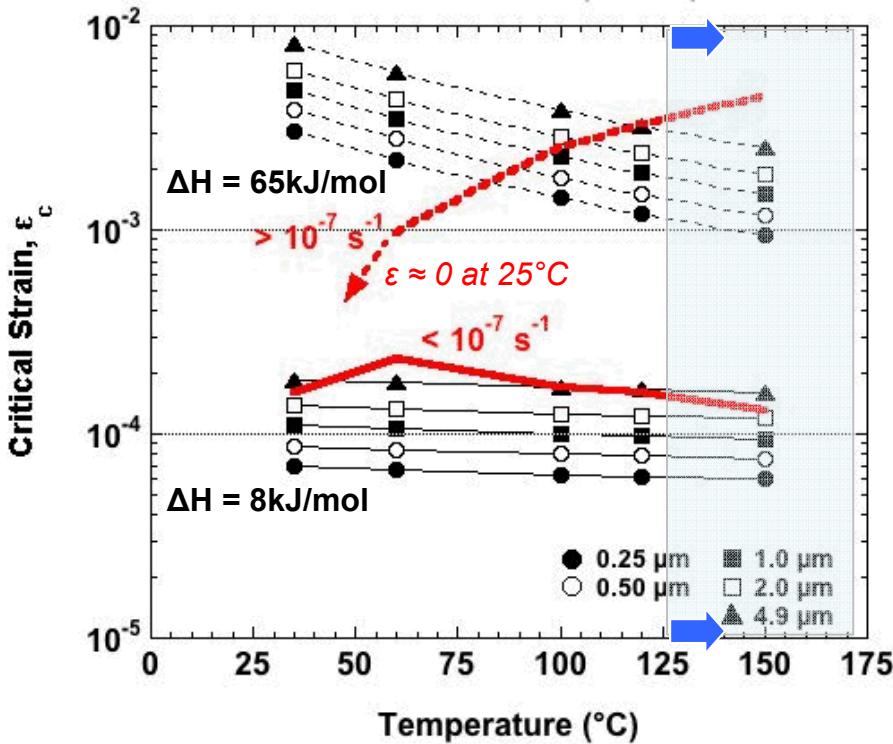


- $d\epsilon/dt > 10^{-7} \text{ s}^{-1} \dots$
- $\epsilon > \epsilon_c, 0.25 - 1.0 \mu\text{m}$
- **Driving force for DRX.**
 - $\epsilon < \epsilon_c$, for 2.0 μm
 - *DRX ... borderline*
- $\epsilon < \epsilon_c, 4.9 \mu\text{m}$
- **No driving force for DRX.**
- $d\epsilon/dt < 10^{-7} \text{ s}^{-1} \dots$
- $\epsilon < \epsilon_c$, for 0.25 – 2.0 μm
- **Driving force for DRX.**
 - $\epsilon \approx \epsilon_c, 4.9 \mu\text{m}$
 - **No driving force for DRX.**

Validation of the DRX-based Model



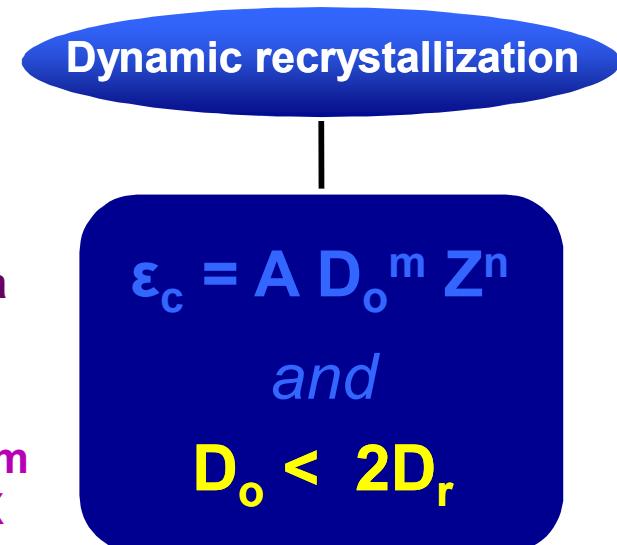
$T > 125^\circ\text{C}$



- $d\epsilon/dt > 10^{-7} \text{ s}^{-1} \dots$
 - $\epsilon < \epsilon_c$, $0.25 - 4.9 \text{ mm}$
 - **Driving force for DRX.**
- $d\epsilon/dt < 10^{-7} \text{ s}^{-1} \dots$
 - $\epsilon < \epsilon_c$, for $0.25 - 1.0 \mu\text{m}$
 - **Driving force for DRX.**
 - $\epsilon < > \epsilon_c$, for $2.0 \mu\text{m}$
 - **DRX ... borderline**
 - $\epsilon < \epsilon_c$, $4.9 \mu\text{m}$
 - **No driving force for DRX.**

Validation of the DRX-based Model

- ◆ *What about ???”*
- ◆ The analysis, $\varepsilon_c = A D_o^m Z^n$, predicted that there was a nominal driving force for DRX in 2.0 μm films in $80^\circ\text{C} < T < 125^\circ\text{C}$.
- ◆ Short-stubby whiskers were observed, indicating continuous DRX rather than cyclic DRX.
 - Long-whisker had diameters of 1.0 – 1.5 μm .
 - $2D_r = 2.0 – 3.0 \mu\text{m}$
 - $D_o < 2.0 – 3.0 \mu\text{m}$
 - Therefore, the 2.0 μm films have too large of a grain size to experience the degree of cyclic DRX required to produce long whiskers.
 - Rather, under the driving force, the 2.0 μm films exhibit the limited, continuous DRX that leads to short, stubby whiskers.
- ◆ The 4.9 μm films are predicted to show no DRX in this temperature range – the uncertainty allows for isolated, continuous DRX.

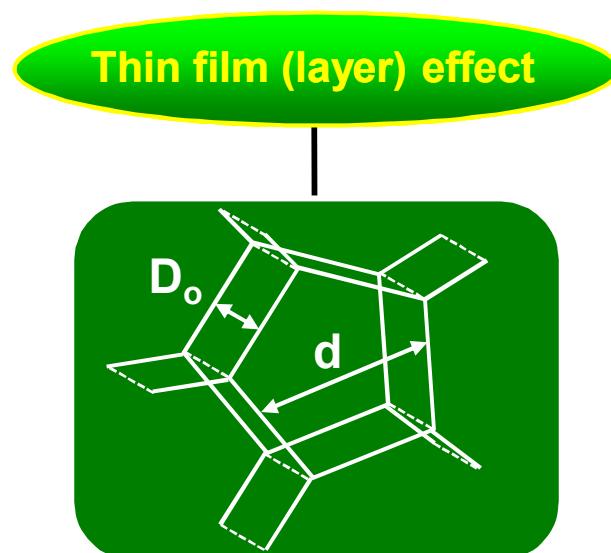
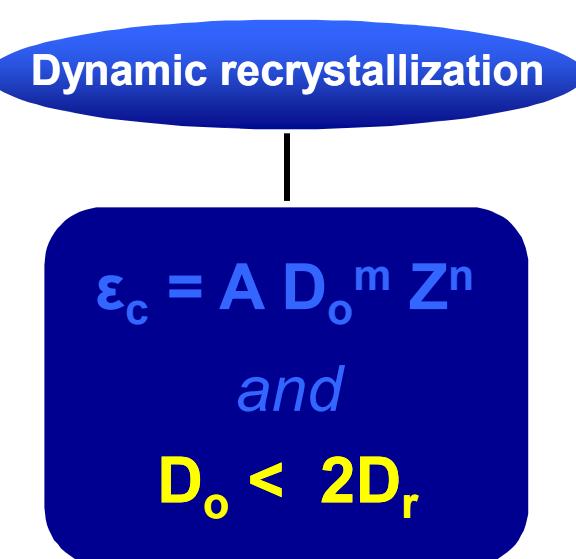


Validation of the DRX-based Model

- ◆ **Hillocks were not observed on 2.0 μm films at all temperature.**

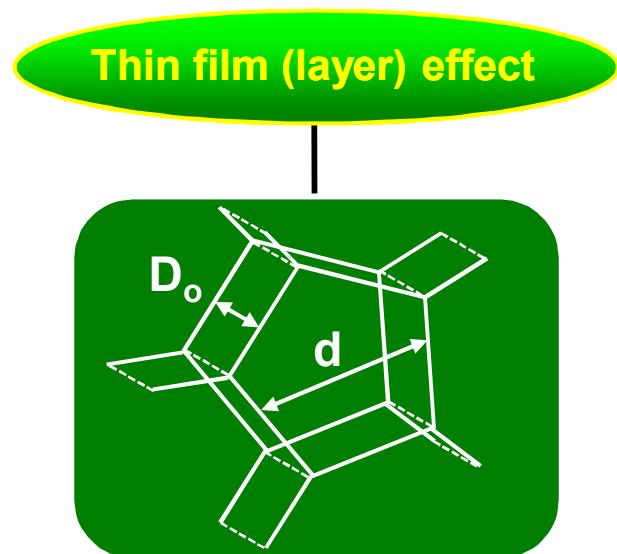
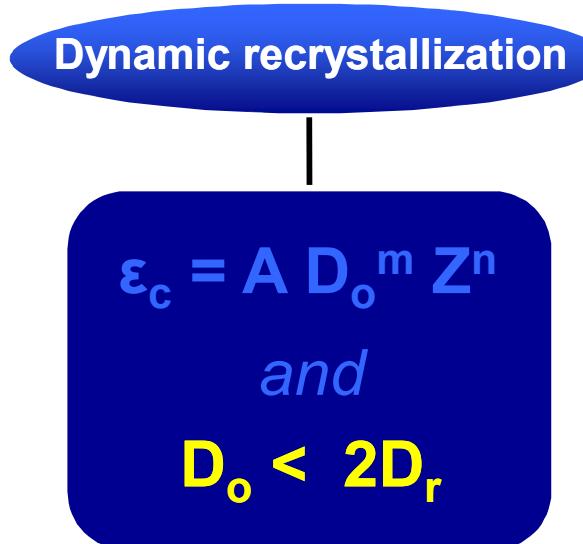
- **Hillocks had diameters of 7.0 – 10 μm .**
- $2D_r = 14 - 20 \mu\text{m}$
- $D_o < 14 - 20 \mu\text{m}$
 - The 2.0 μm films have a $D_o < 14 - 20 \mu\text{m}$ so hillocks would be expected on the surface.

- **Why aren't there any hillocks ?**
- **Thin film effect:**
 - $D_o \approx \text{or} < d$, grain boundaries **appear pinned**.
 - $2.0 \mu\text{m}$... *versus* ... $1 - 5 \mu\text{m}$ 
 - Since D_o is comparable to d , grain boundaries are pinned, which prevents hillocks.



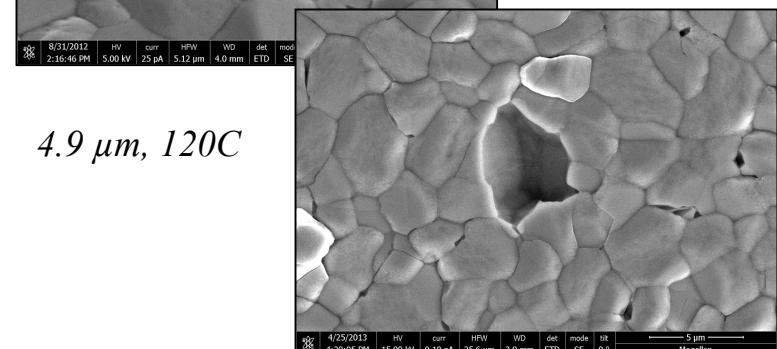
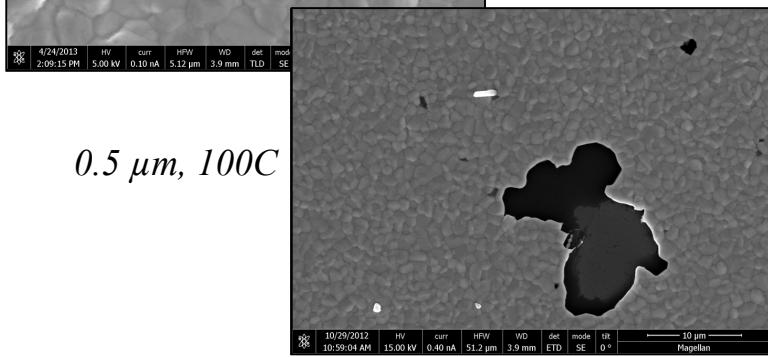
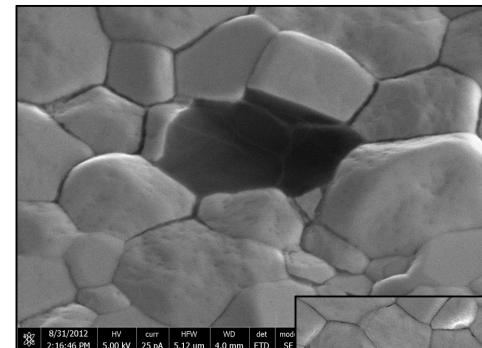
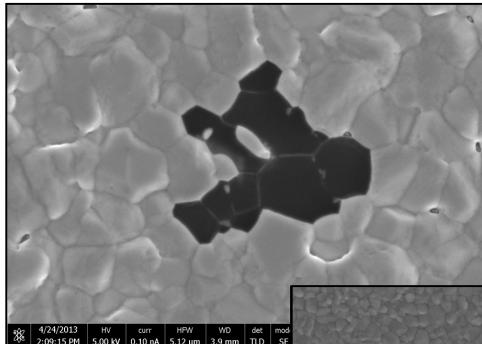
Validation of the DRX-based Model

- ◆ The analysis, $\varepsilon_c = A D_o^m Z^n$, predicted that there was a driving force for DRX in 4.9 μm films at $T > 125^\circ\text{C}$ (in the regime, $> 10^{-7} \text{ s}^{-1}$).
 - $D_o < 2.0 - 3.0 \mu\text{m}$ for long whiskers
 - Therefore, the 4.9 μm grain size was too large to support the cyclic DRX required for long whiskers. *There were a few, short, stubby whiskers formed by continuous DRX.*
 - $D_o < 14 - 20 \mu\text{m}$ for hillocks
 - Therefore, the 4.9 μm films can potentially experience hillock growth.
 - Thin film effect:
 - $D_o > d$, grain boundary pinning is unlikely.
 - $4.9 \mu\text{m}$... *versus* ... $< 1 \mu\text{m}$ to $3 - 4 \mu\text{m}$
 - Since $D_o > d$, grain boundaries are *not* pinned, which encourages the formation of hillocks.



Depleted Zones

- ◆ The size of depleted zones was a function of the Sn film thickness.



$$D_o \leq 1.0 \mu\text{m}$$

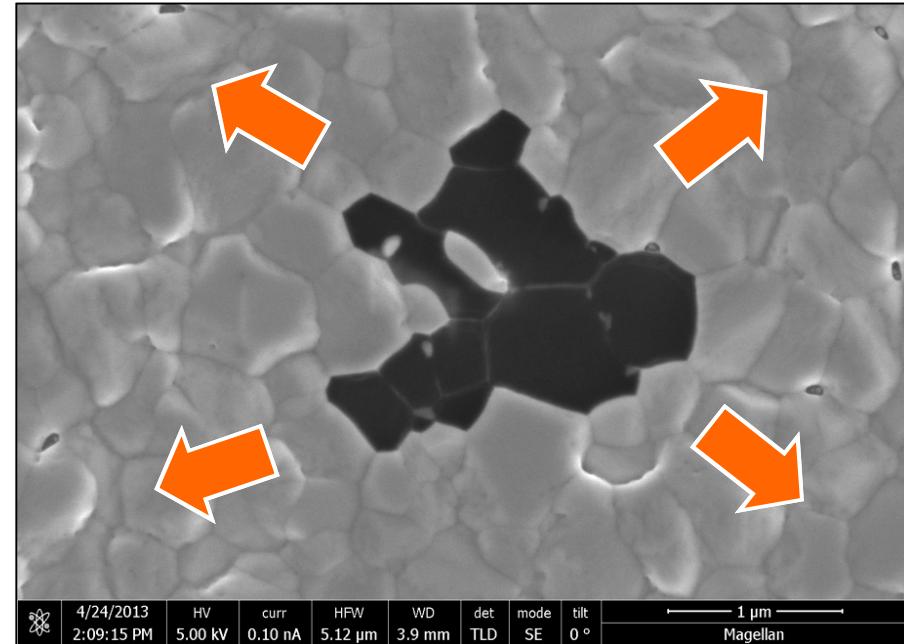
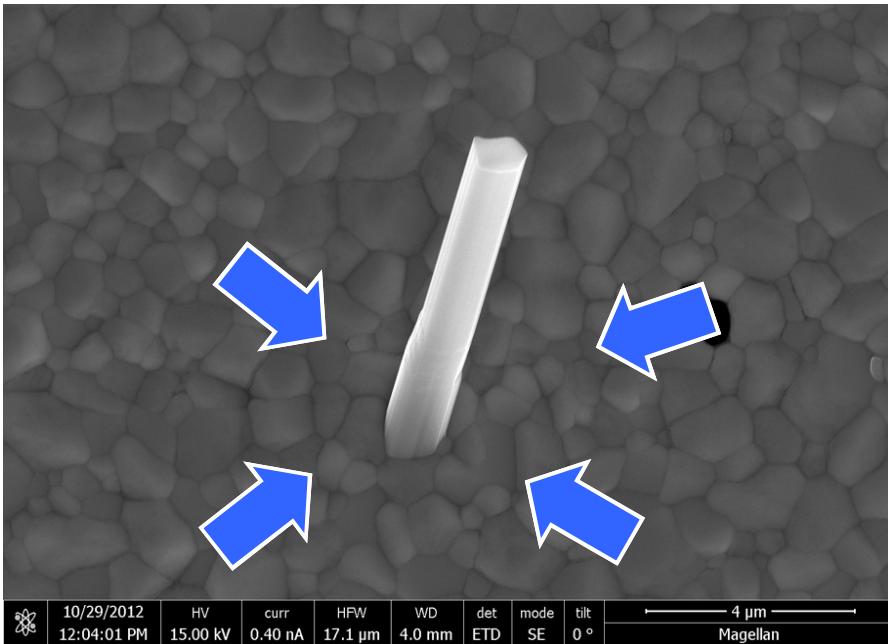
Few, but larger depleted zones.

$$D_o > 1.0 \mu\text{m}$$

Numerous, but small depleted zones.

Depleted Zones

- Unlike whiskers where atoms diffuse towards a local area ... depleted zones result material diffusion away from the area.



- Also, unlike whiskers ...
 - Depleted zones do not require DRX.
 - The driving force is a tensile stress.
- Like whiskers, depleted zone requires long-range diffusion.

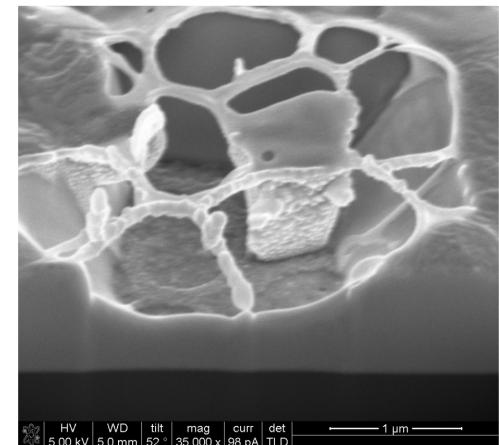
Depleted Zones

- ◆ The depleted zones are also controlled by the local stress field.

Film Thickness	Temperature (C)				
	35	60	100	120	150
0.25	C T N	C T N	C T N	C T N	C T N
0.5	C T N	C T N	C T N	C T N	C T N
1.0	C T N	C T N	C T N	C T N	C T N
2.0	C T N	C T N	C T N	C T N	C T N
4.9	C T N	C T N	C T N	C T N	C T N

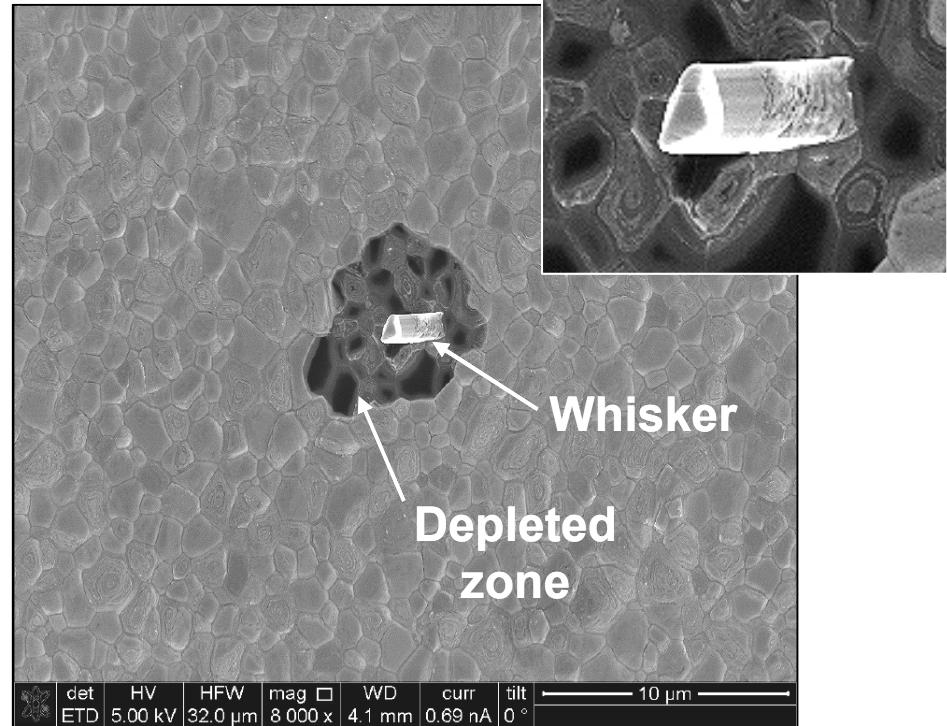
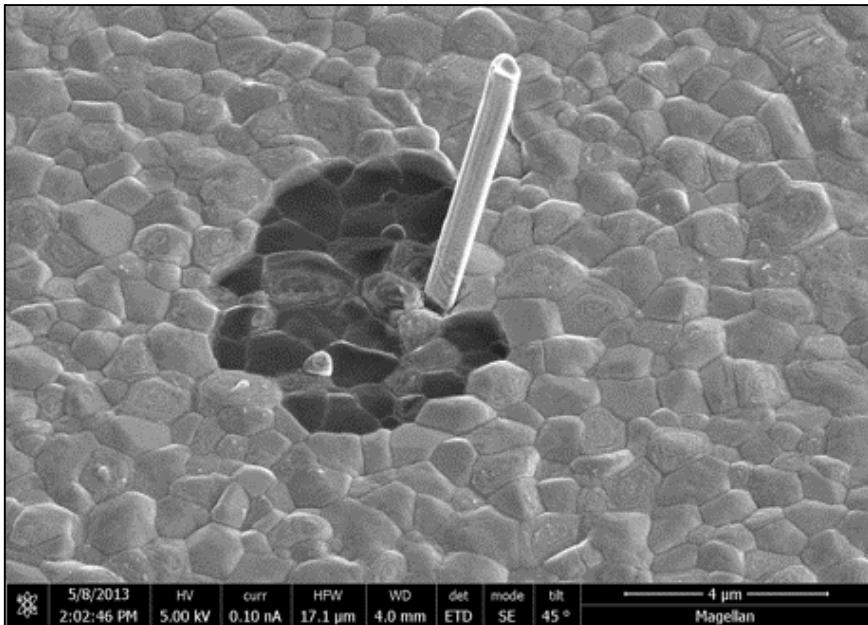
Green is present; red is absent. "C," compression; "T," tension; and "N," no-load

- Depleted zones do not show a sensitivity to the Sn/Si CTE mismatch compressive stress at elevated temperatures.
- The applied load did not have an effect on the propensity of depleted zones.
- The decrease of depleted zone activity with lower temperatures of 60°C and 30°C reflects a slowing of the long-range diffusion process.
 - This scenario similarly applies to the long whisker and hillock data discussed earlier.



Depleted Zones

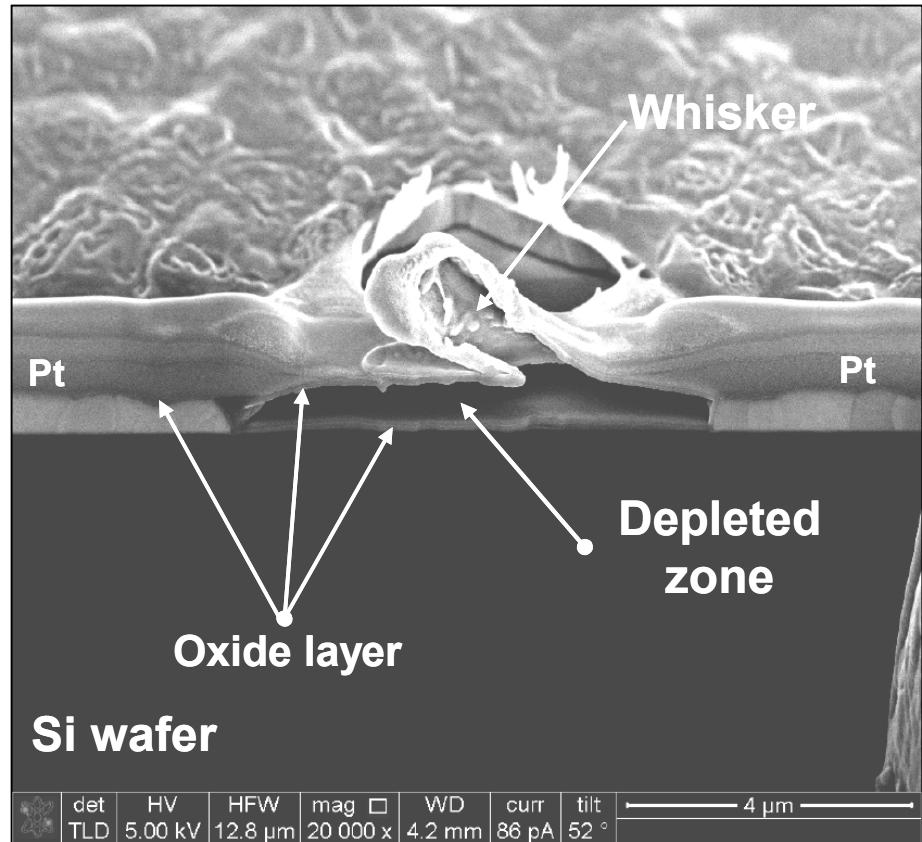
- ◆ An important phenomenon observed in this study was **whiskers associated with depleted zones**.
- ◆ This behavior was unique to:
0.5 μm films ... 150°C (9 days) ... and *any load*.



Depleted Zones

- ◆ Moreover, those Sn whiskers were found to be **hollow**.
- ◆ **Stress reversal scenario:**

- The whisker forms under the compressive stress.
- Simultaneously, there is formation of the oxide layer.
- There is the reversal of the stress to tension.
- Under the tensile stress, the Sn atoms diffuse away, leaving the hollow whisker and “covered” depleted zone.



- ◆ Stress reversals are not uncommon in thin films. And, such reversals are sensitive to film thickness – 0.5 μm case, here.

Summary

- ◆ This study validated **cyclic dynamic recrystallization (DRX)** as the controlling mechanism responsible for the development of long whiskers and hillocks.
- ◆ The supporting mechanism is **long-range diffusion**.
- ◆ The thin film nature of the Sn layer also has a role in whisker development – **specimen thickness effect** (“*d* vs. *h*”).
- ◆ The particular susceptibility of Sn to long whiskers is due to the **bi-modal creep mechanism** present at the relevant temperature and stress conditions.
- ◆ **Continuous DRX** appeared in the form of short, stubby whiskers.
- ◆ Although **depleted zones** do not require DRX, their trends provided insight into the **long-range diffusion rate kinetics**.

