

Sinter Monitoring and Control of Lunar Soil Simulant and its application to Asteroid Regolith

Gregory Konesky
K-Plasma, Ltd.

3rd Joint SRR/PTMSS

Colorado School of Mines, Golden, CO

June 4-7, 2012

Potential Sintered Lunar Soil Applications

Roadways, Walkways

Launch Pads, Berms, Dust Control

Foundations, Trench Wall Liners

Radiation/Thermal Shielding

Basic Construction Elements:

Bricks, Columns, Panels, etc.

Methods of In-situ Sintering

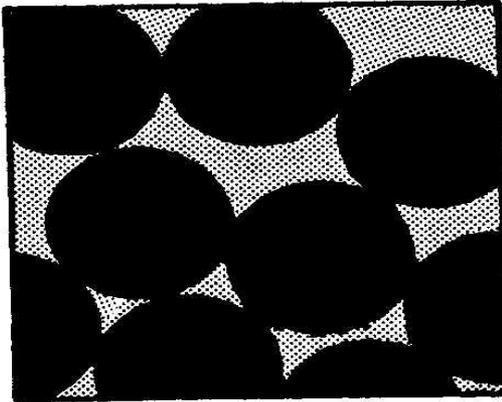
Radiant Heating Elements

Focused Solar Energy

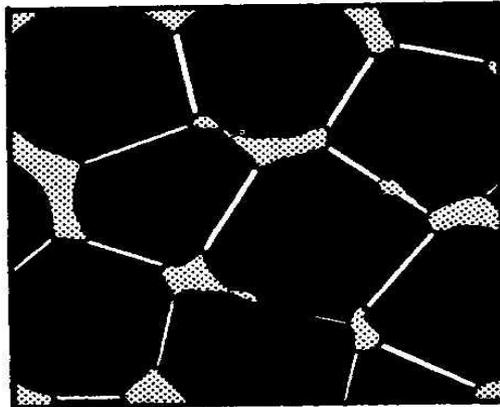
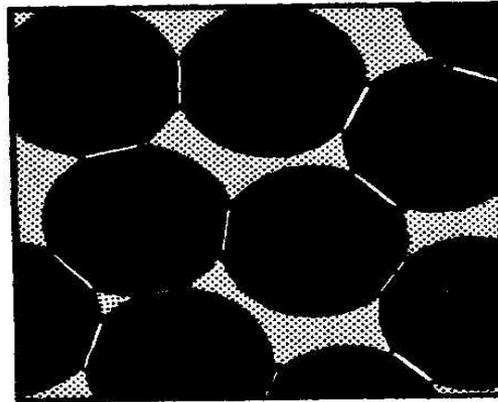
Microwaves

Stages of Sintering

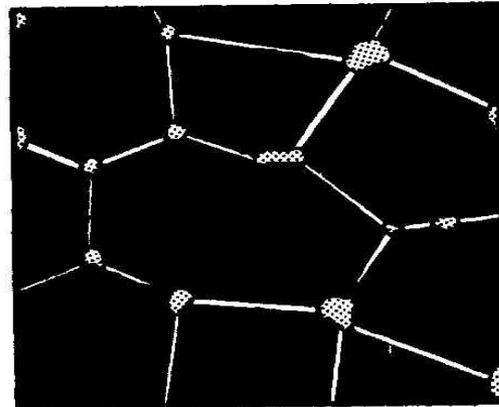
loose powder



initial stage



intermediate stage



final stage

Degree-of-Sintering Issues

Low Degree-of-Sintering

Low Mechanical Strength, High Porosity

Optimum Degree-of-Sintering

Moderate Strength, Somewhat Brittle,
Mildly Porous

High Degree-of-Sintering

High Strength, Very Brittle, Low Porosity

Sinter-Specific Applications

Thermal Shielding

High Porosity, Low Density

Radiation Shielding

High Density, Low porosity

Landing Pads

Good Mechanical Strength, Impact Resistant

Advanced Sintering Approaches

Sinter Profiles, Gradients

High Density Veneer, Low(er) Density Bulk
(Roadways)

Sandwich Construction

High-Low-High Density Profile
(Panels)

Advanced Sintering Approaches

Fibers:

Drawing, Spinning (Electro-spinning?)

Composites:

S/S fibers, CNTs, BN-NTs

Advanced Sintering Approaches

Fibers:

Drawing, Spinning (Electro-spinning?)

Composites:

S/S fibers, CNTs, BN-NTs

Not Just Moon Bricks

Real-Time In-Situ Sinter Monitoring

Processing Temperature Profile:

Largely “Cut and Try”

Dependent on Green Body
Composition and Particle Size Distribution

Dependent on Green Body
Size and Shape

Real-Time In-Situ Sinter Monitoring

Goal:

To develop a Monitoring Approach

Simple

Low Cost

Fast

To Detect Desired End-Point Signature

To Know When To Stop Heating

Real-Time In-Situ Sinter Monitoring Approaches

Changes in Properties

Density

Interaction with Penetrating Radiation

Acoustical

Interaction with Electromagnetic Waves

Electrical Conductivity Properties

Must be Compatible with High temperatures

≈1100°C

Real-Time In-Situ Sinter Monitoring Approaches

Impedance Spectroscopy

Analyze Sample with low-level
Frequency Variable Signal

Plot Real and Imaginary Components (Bode Plot)
over a range of Frequencies

Derive First Order Approximation of
Grain Size and Boundary Characteristics

Real-Time In-Situ Sinter Monitoring Approaches

Impedance Spectroscopy Issues

Complex

Slow

Process May Change Too Fast

Real-Time In-Situ Sinter Monitoring Approaches

Impedance Monitoring of a Single Frequency

Simple

Fast

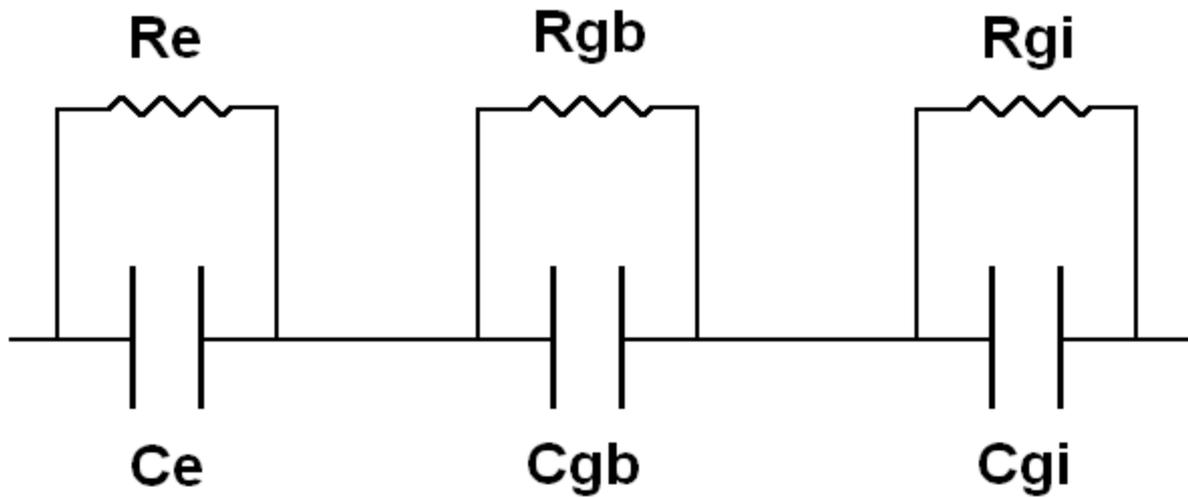
Can be Selected to Monitor:

Changes in Capacitance

Changes in Ionic Conductivity

or Both

Equivalent Circuit

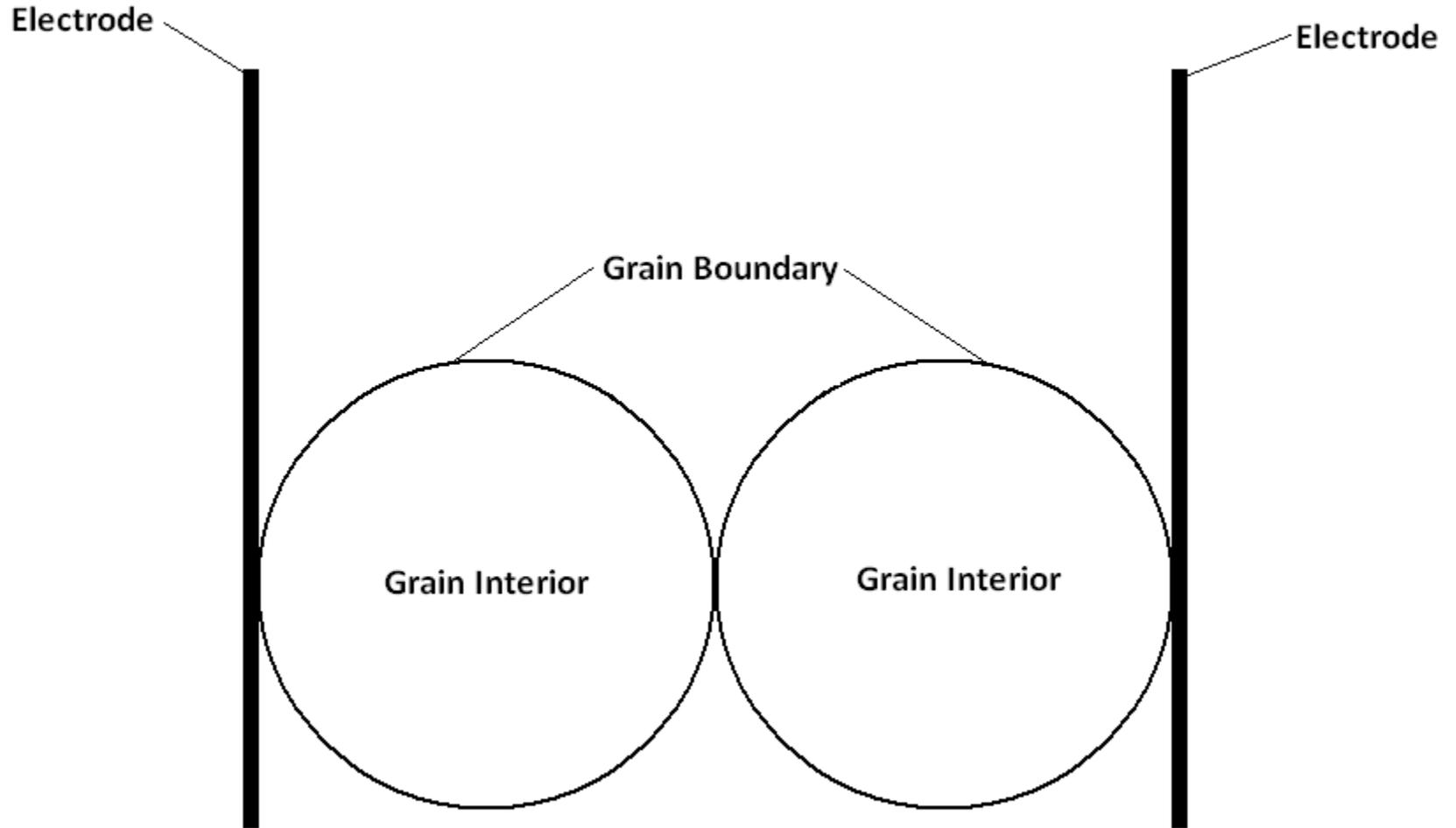


e = electrode

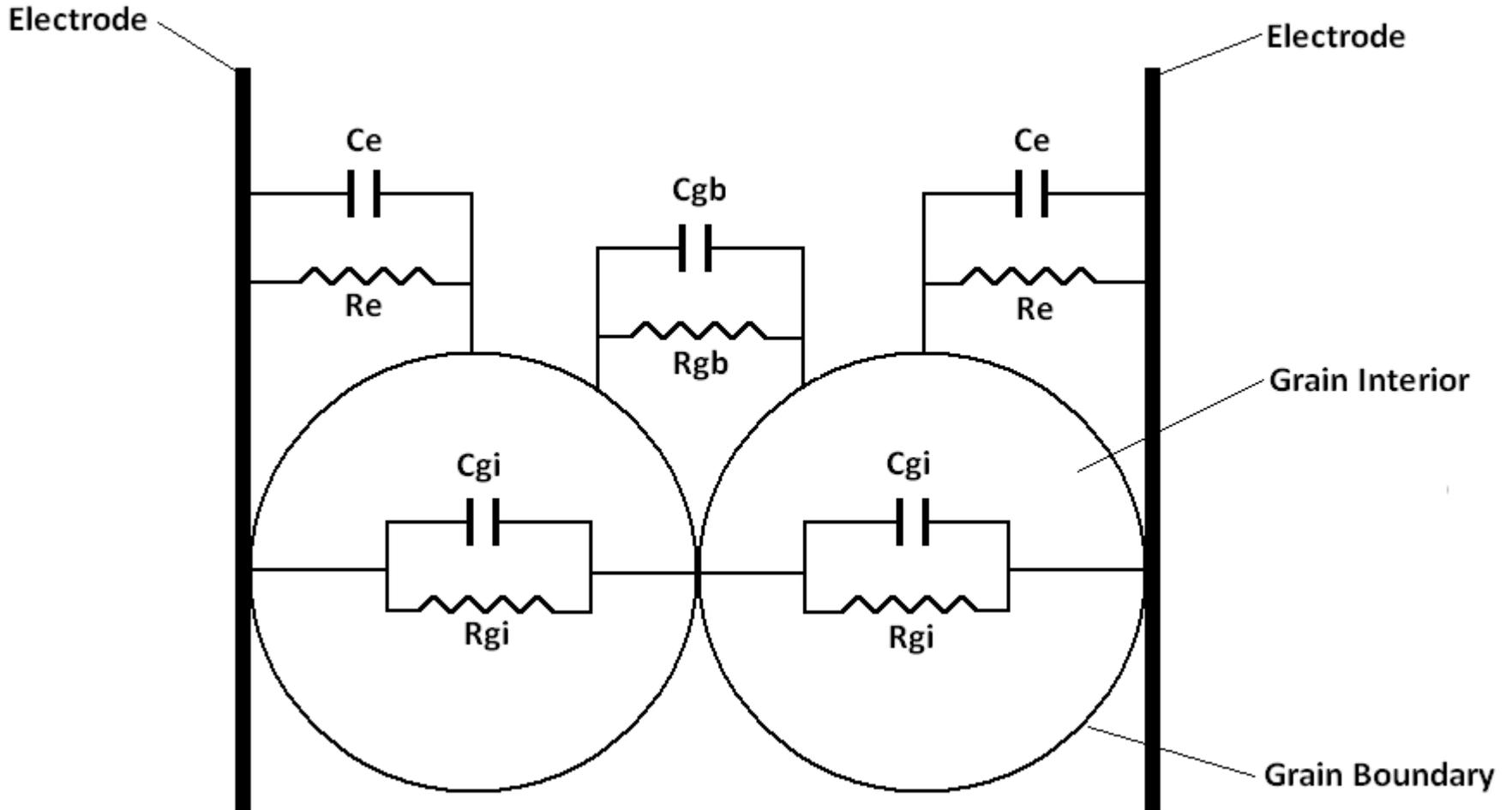
gb = grain boundary

gi = grain interior

Equivalent Circuit



Equivalent Circuit



Separating Capacitive and Resistive Components

Capacitive Component:

$$X_C = 1 / \omega C = 1 / 2 \pi f C$$

Resistive Component:

$R_x =$ Ionic Conductivity

Arrhenius – like Behavior

Temperature Dependence

Resistive Conduction Components

$$\sigma = \sum c_i q_i \mu_i + n_e q_e \mu_e + n_h q_h \mu_h$$

where:

c_i is the **concentration of ionic defects**

n_e, n_h are the **electron and hole concentration**

q_i, q_e, q_h are the **ion, electron, and hole charge**

μ_i, μ_e, μ_h are the **ion, electron, and hole mobility**

Arrhenius Equation

$$\sigma = A/T e^{-E_a/K_b T}$$

where:

σ is the **Conductivity**

A is the pre-exponential **Constant**

T is the **Temperature**

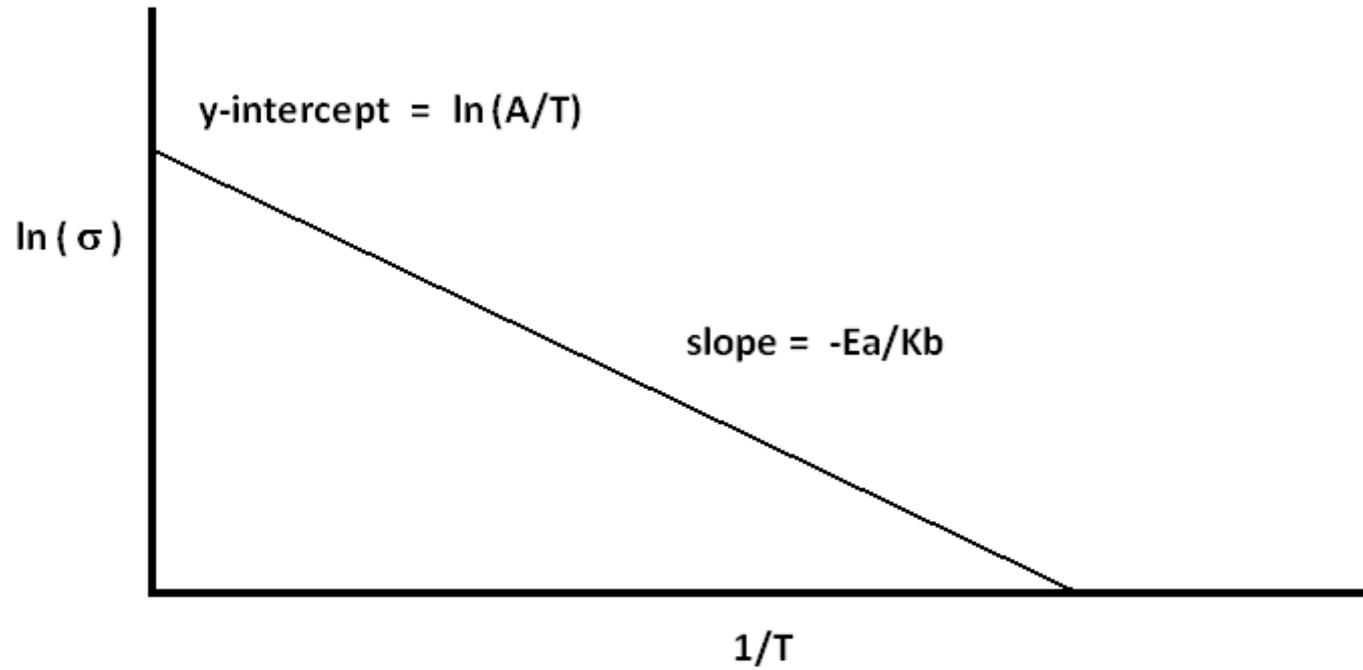
E_a is the **Activation Energy**

K_b is **Boltzmann's Constant**

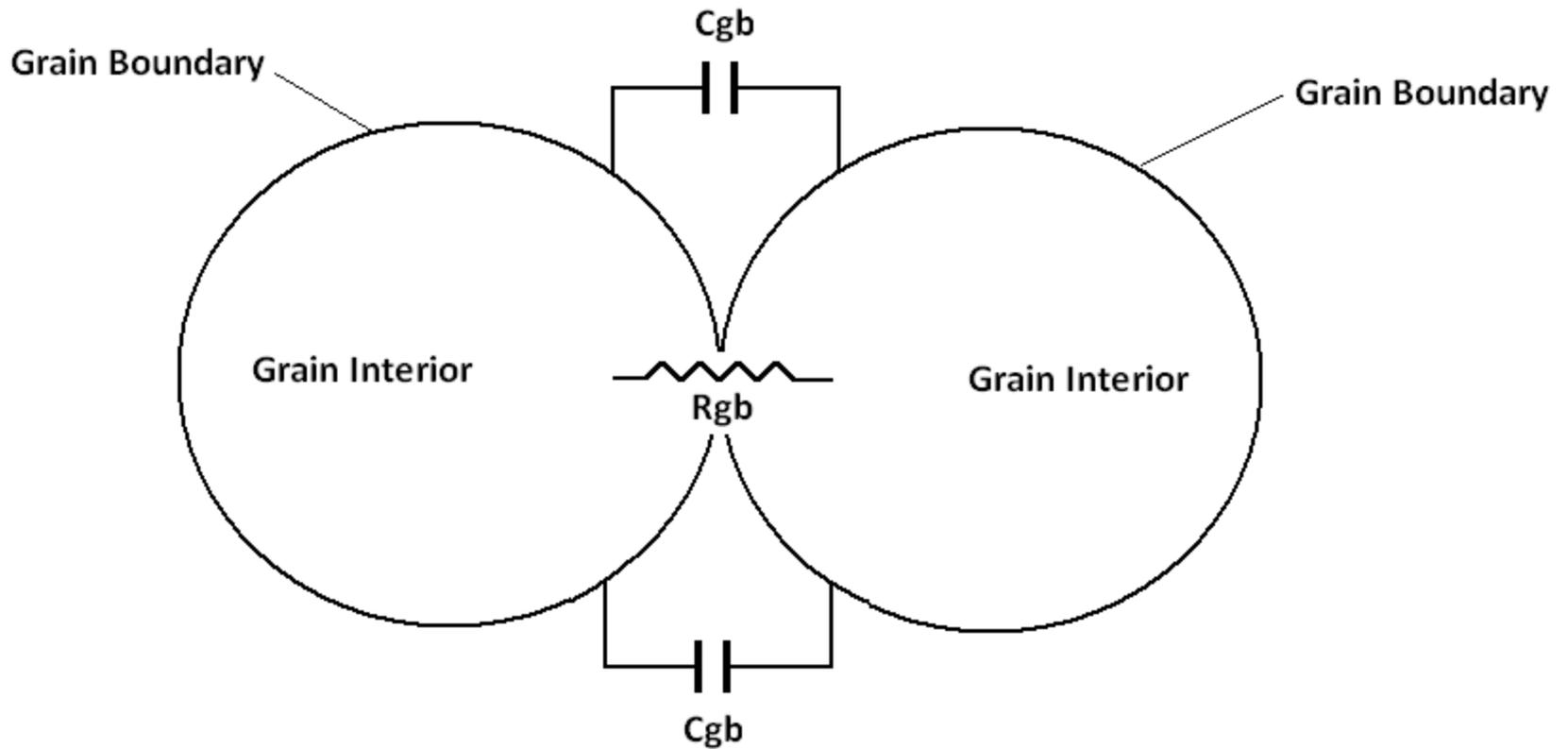
Arrhenius Plot

$$\ln(\sigma) = \ln(A/T) - (E_a/K_b) (1/T)$$

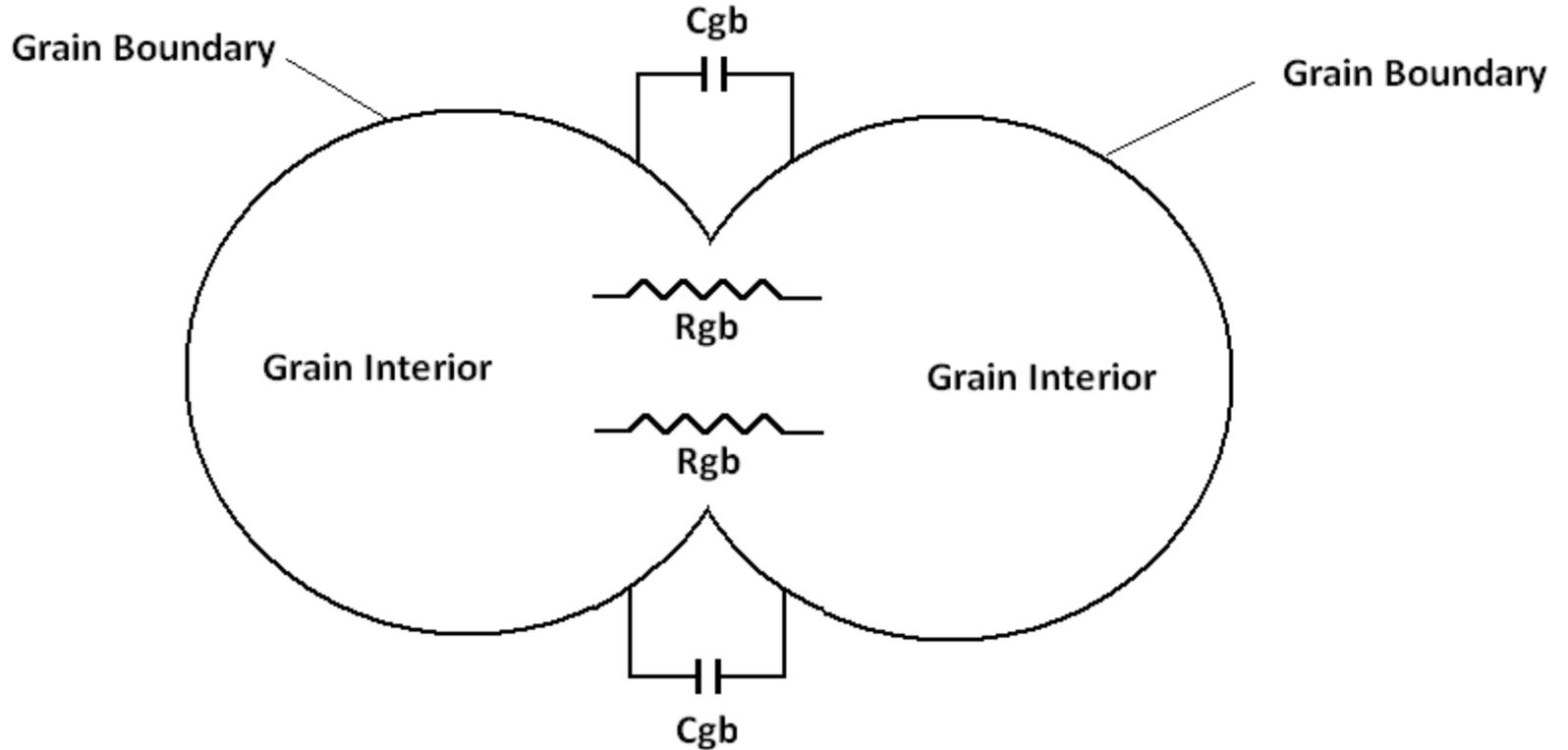
Arrhenius Plot



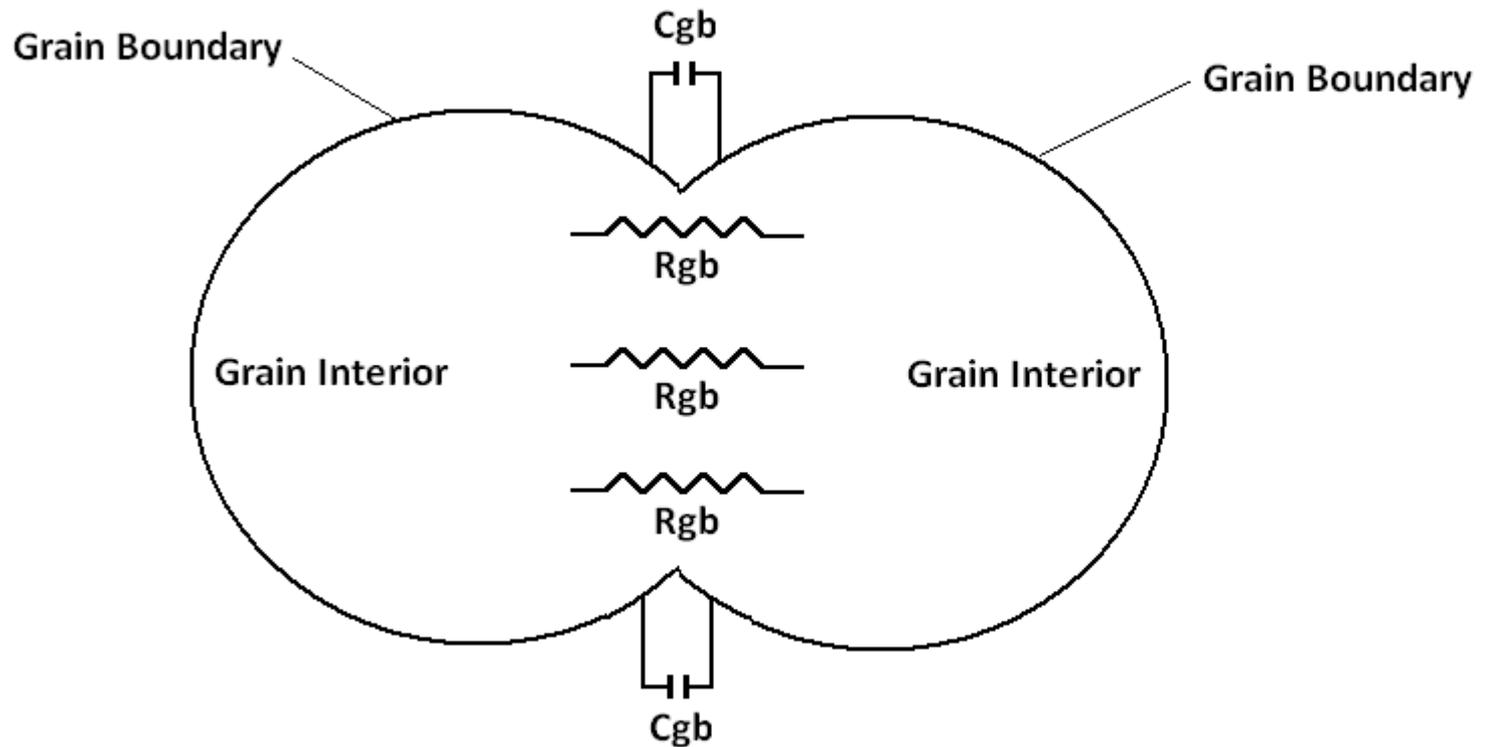
Loose Powder Equivalent Circuit



Initial Sintering Equivalent Circuit



Intermediate Sintering Equivalent Circuit



Sintering Endpoint Detection Signature

No Grain Boundary Melting:

Simple Arrhenius-like Temperature Dependence

Onset of sintering:

Arrhenius-like Temperature Dependence

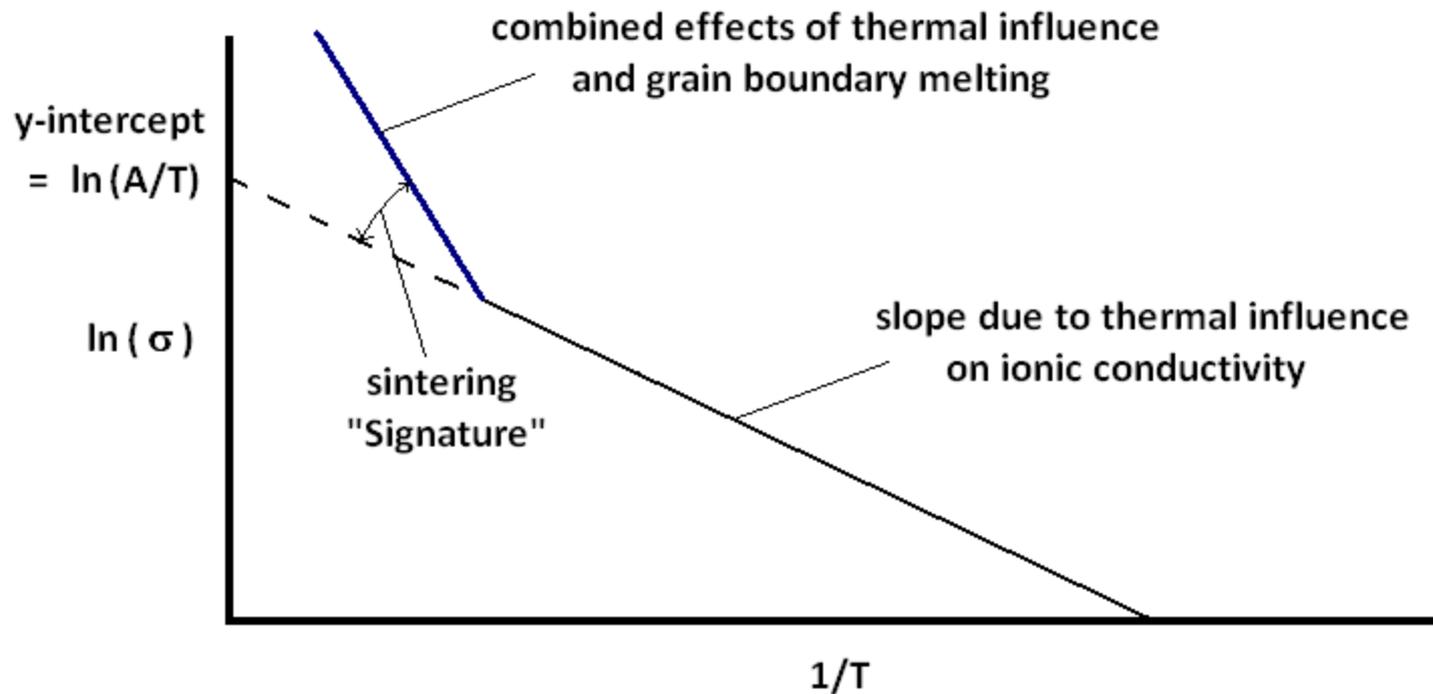
+

Additional Increase in Conductivity

Due to Grain Boundary Melting

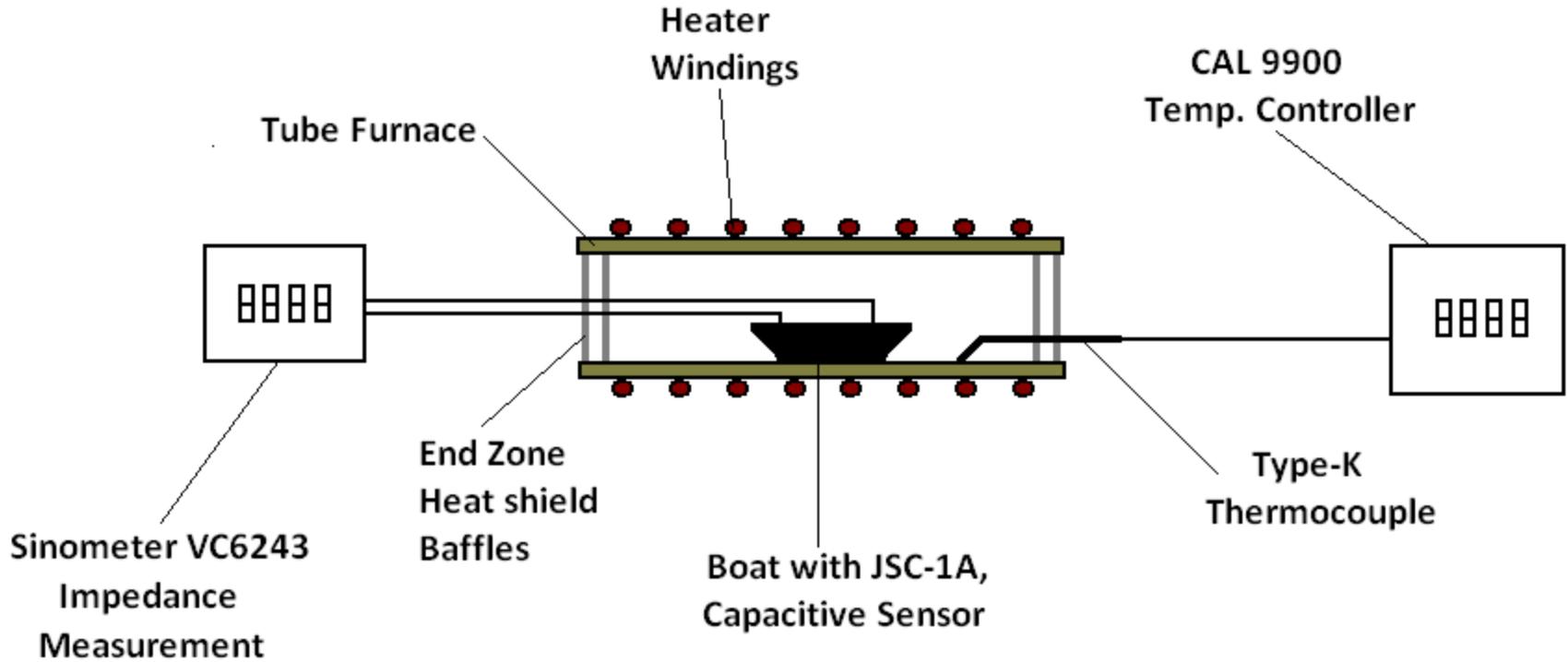
Change in Slope of Arrhenius Plot

Sintering Endpoint Detection Signature



The Experiment

Basic Setup



Combustion Boat and Capacitive Sensor



20 ml High-Alumina Boat

Capacitive Sensor:

304 S/S 75mm long, 12mm wide, 0.1mm thick

Insulators: 6.4mm dia., 19 mm long

Combustion Boat and Capacitive Sensor

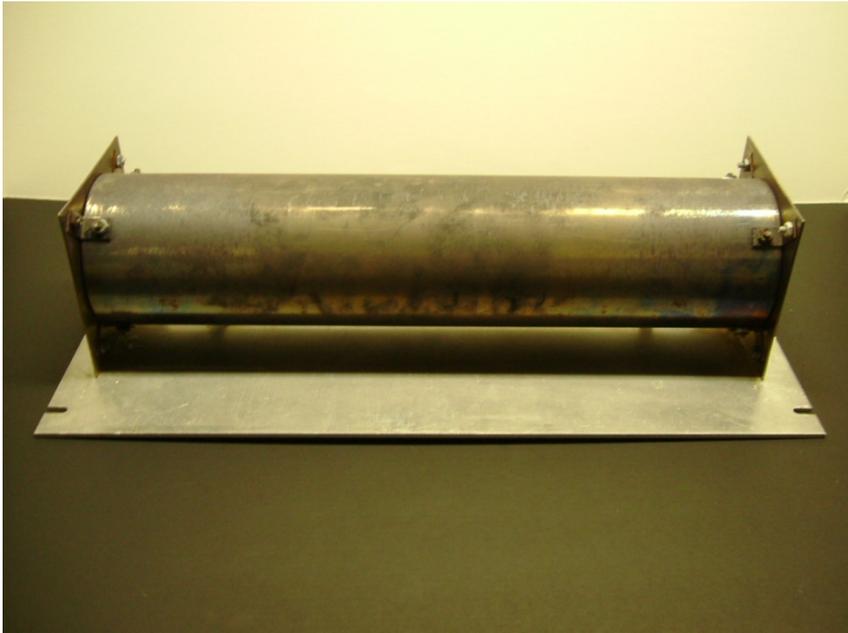


Wire Leads: 304 S/S
0.5mm dia.



Typical JSC-1A Charge:
28.4 grams

Tube Furnace

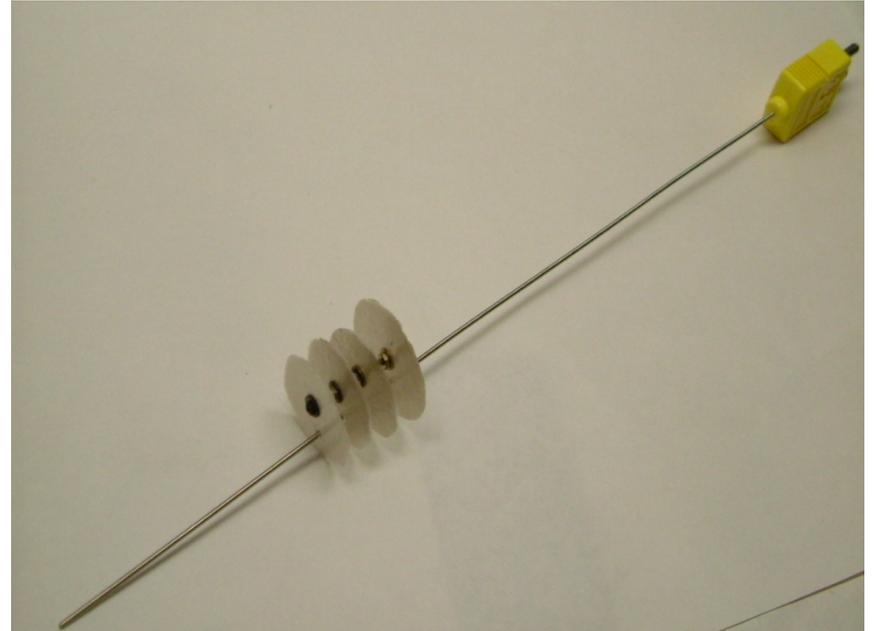
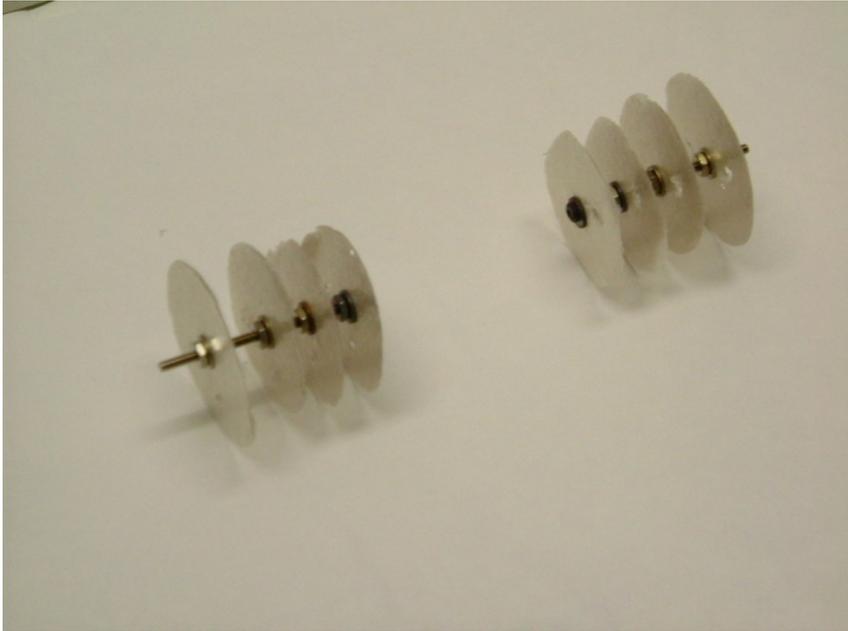


Mullite Tube
457mm (18") long
31.8mm (1.25") ID
Kanthal A1 Heater Wire



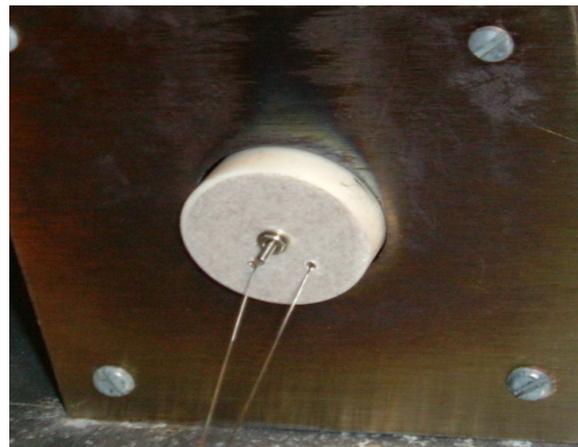
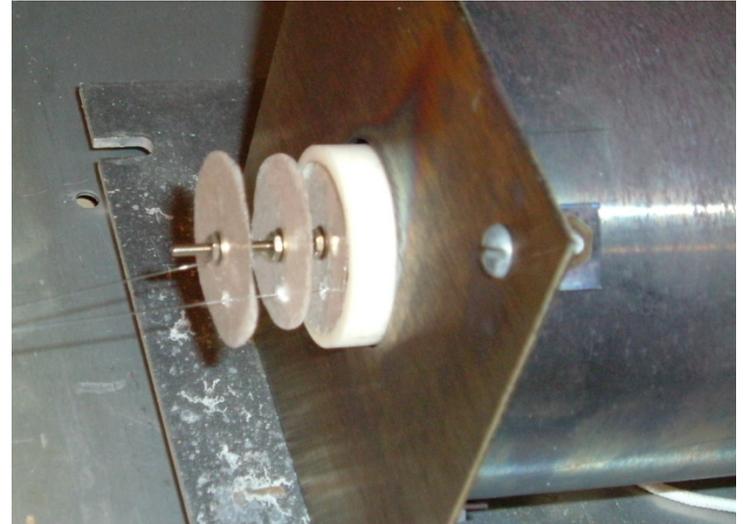
305mm (12") Hot Zone
 $\pm 2^{\circ}\text{C}$ Uniformity over
Central 203mm (8") Zone
at 1160°C

End-Zone Heat Shield Baffles

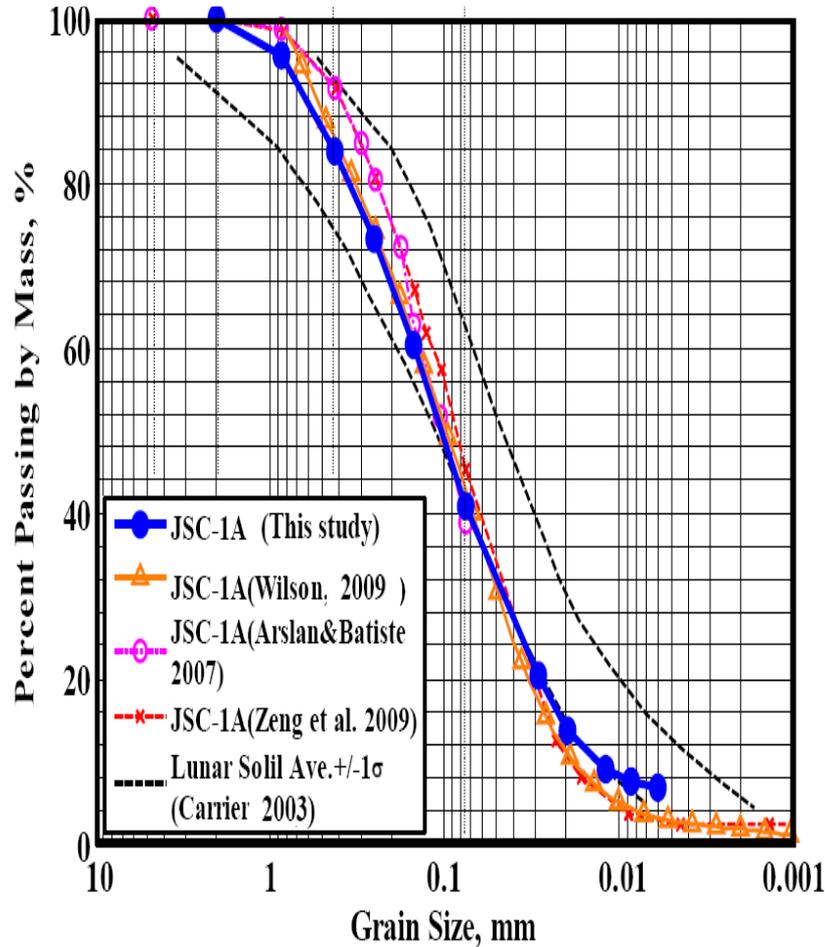


Mica Heat Shield Baffles

Loading Tube Furnace



Lunar Soil Simulant JSC-1A

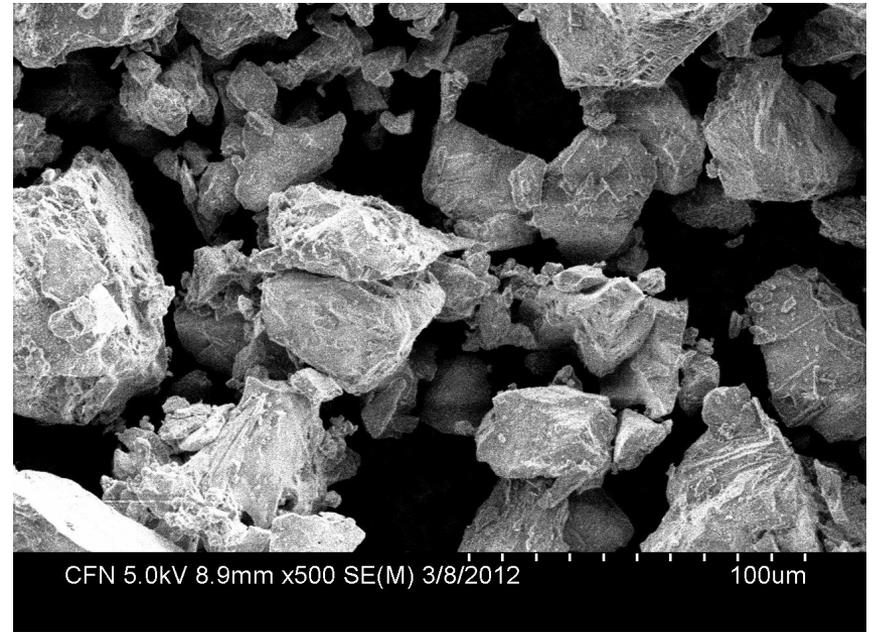
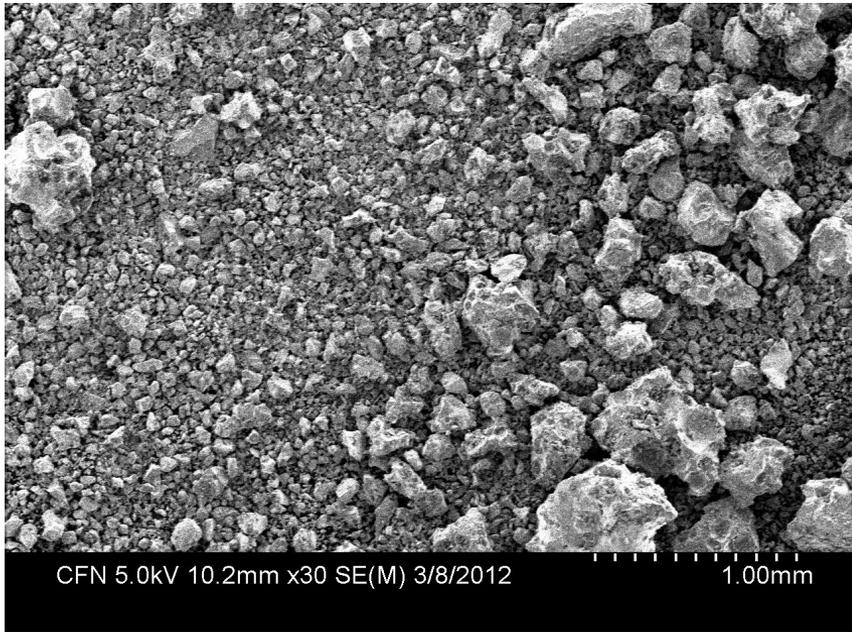


lai, 2010

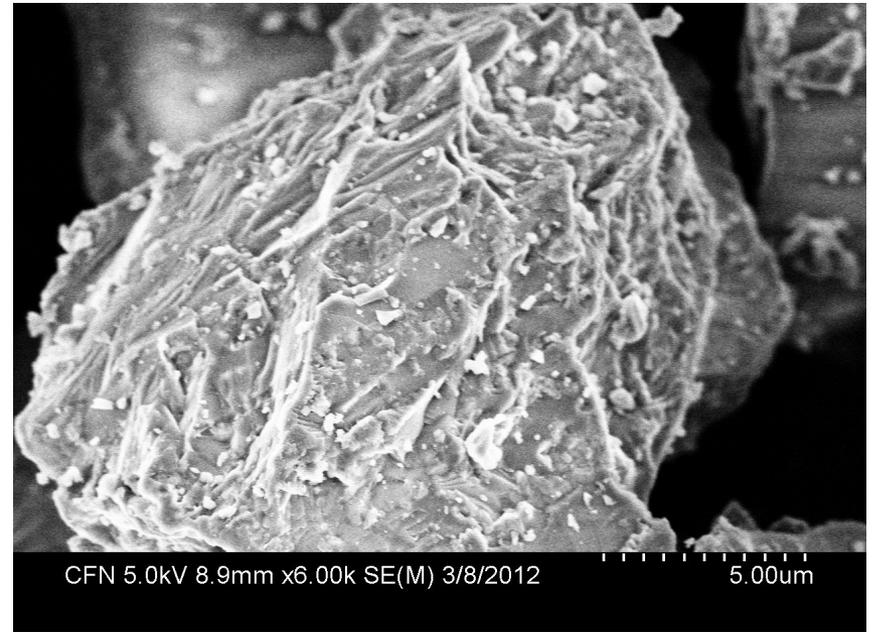
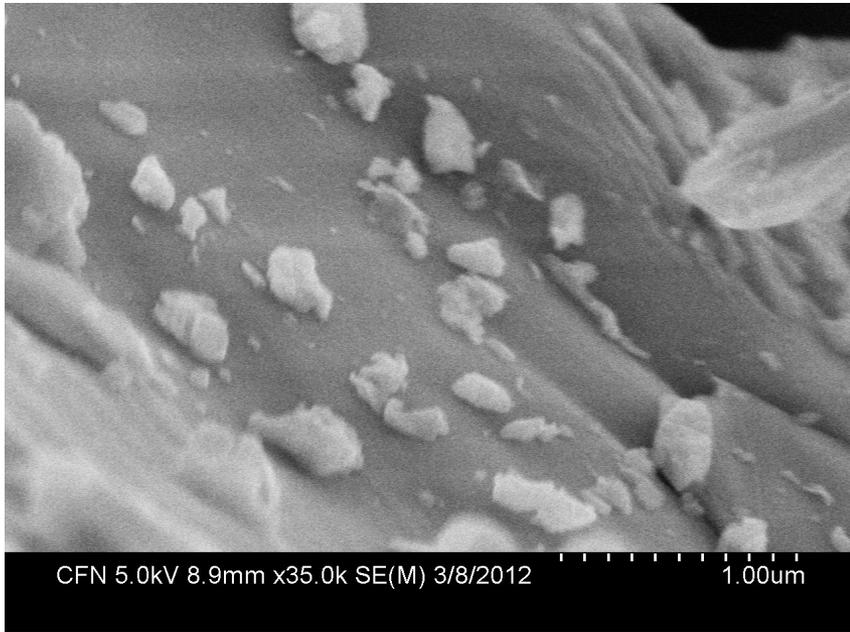
Oxide	JSC-1 (mean of 3)		Lunar Soil 14163*
	Conc.	Std. Dev.	Conc.
	Wt %	Wt %	Wt %
SiO ₂	47.71	0.10	47.3
TiO ₂	1.59	0.01	1.6
Al ₂ O ₃	15.02	0.04	17.8
Fe ₂ O ₃	3.44	0.03	0.0
FeO	7.35	0.05	10.5
MgO	9.01	0.09	9.6
CaO	10.42	0.03	11.4
Na ₂ O	2.70	0.03	0.7
K ₂ O	0.82	0.02	0.6
MnO	0.18	0.00	0.1
Cr ₂ O ₃	0.04	0.00	0.2
P ₂ O ₅	0.66	0.01	---
LOI	0.71	0.05	---
Total	99.65		99.8

Papike, et al, 1982

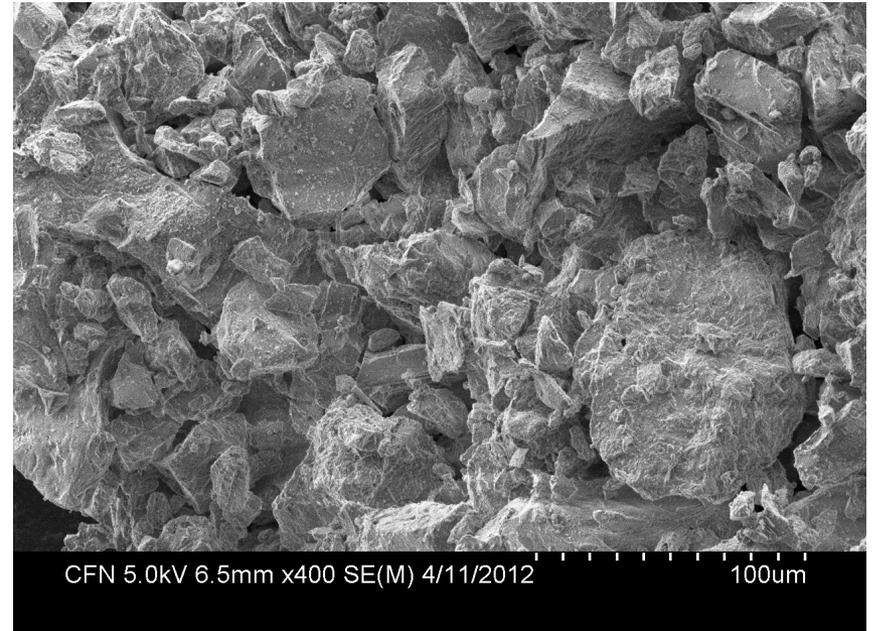
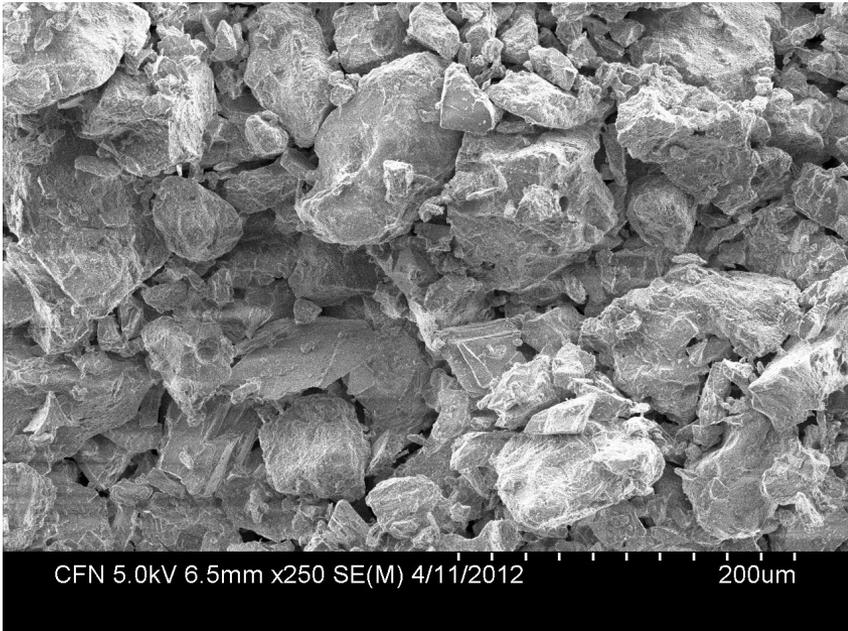
As-Received JSC-1A



As-Received JSC-1A



Minimally Sintered

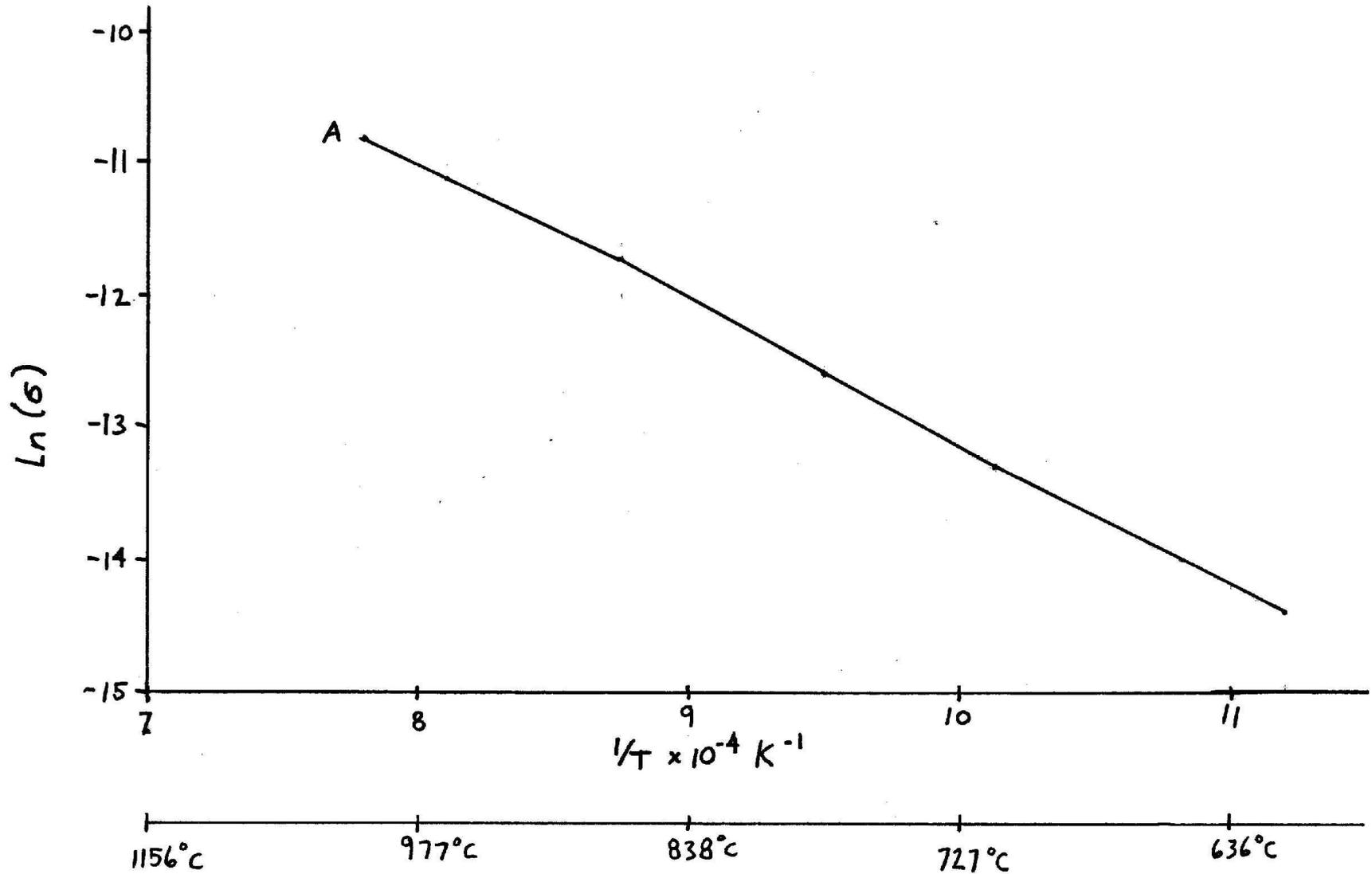


1.5 Hrs. at $\sim 1000^{\circ}\text{C}$

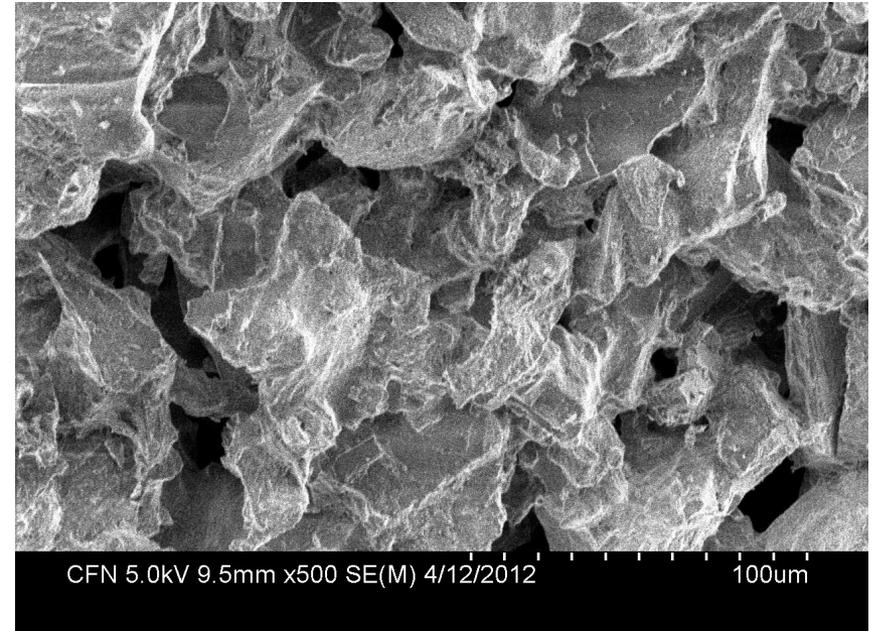
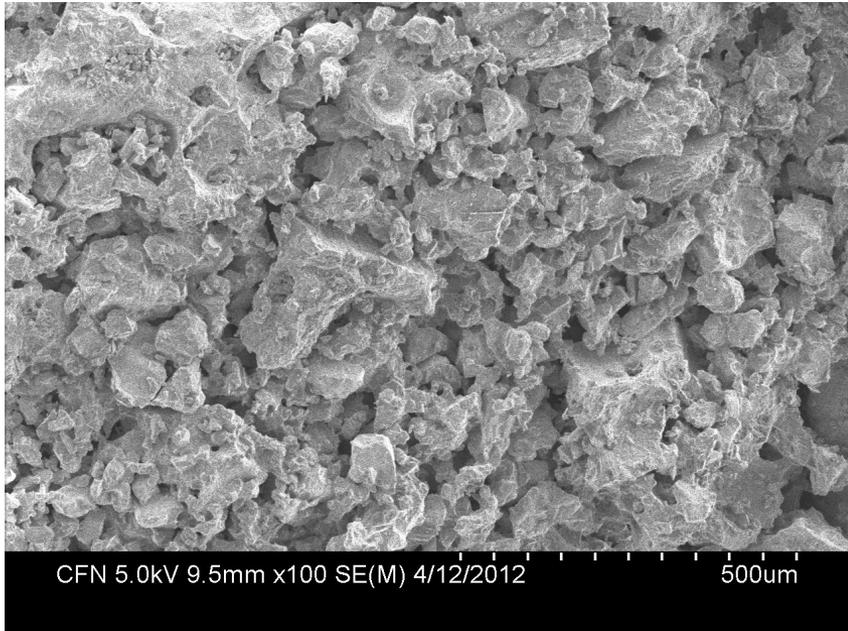
Minimally Sintered



Minimally Sintered



Further Increase in Sintering



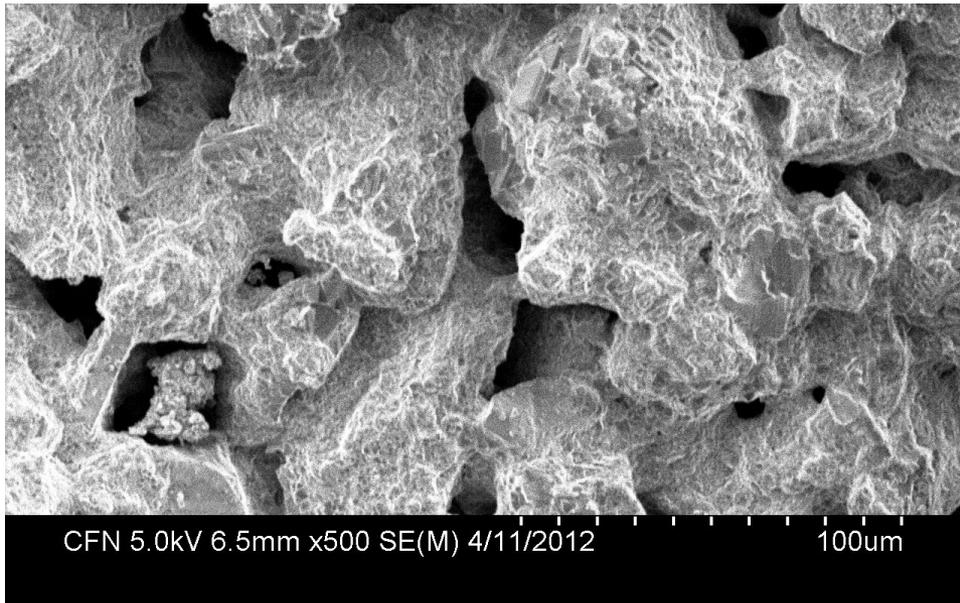
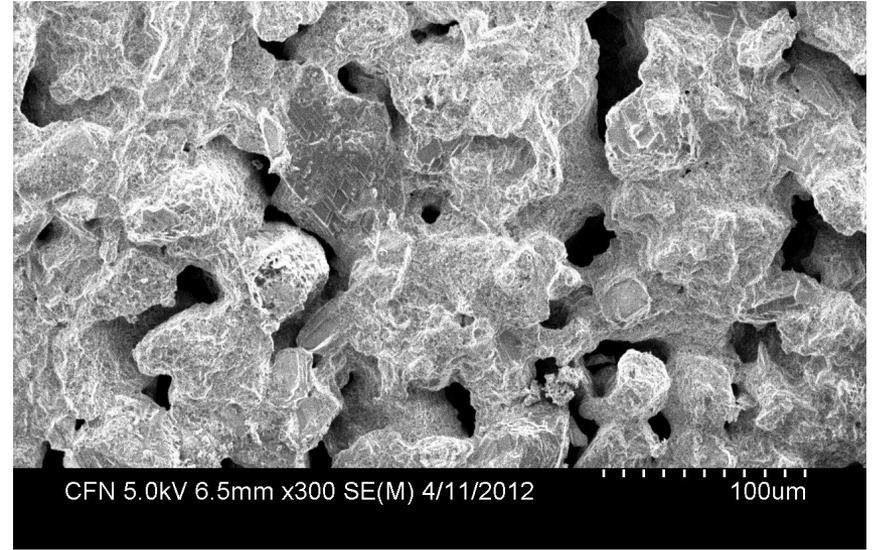
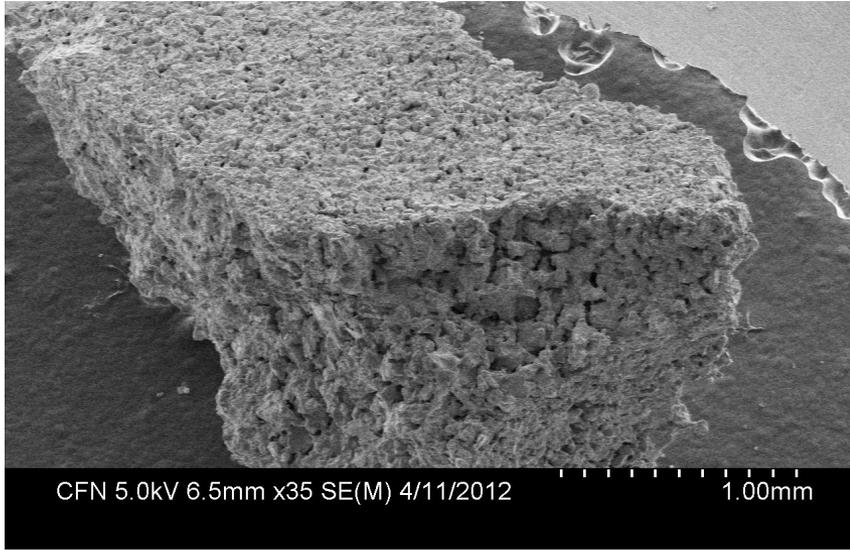
1.5 Hrs. at $\sim 1075^{\circ}\text{C}$

Further Increase in Sintering



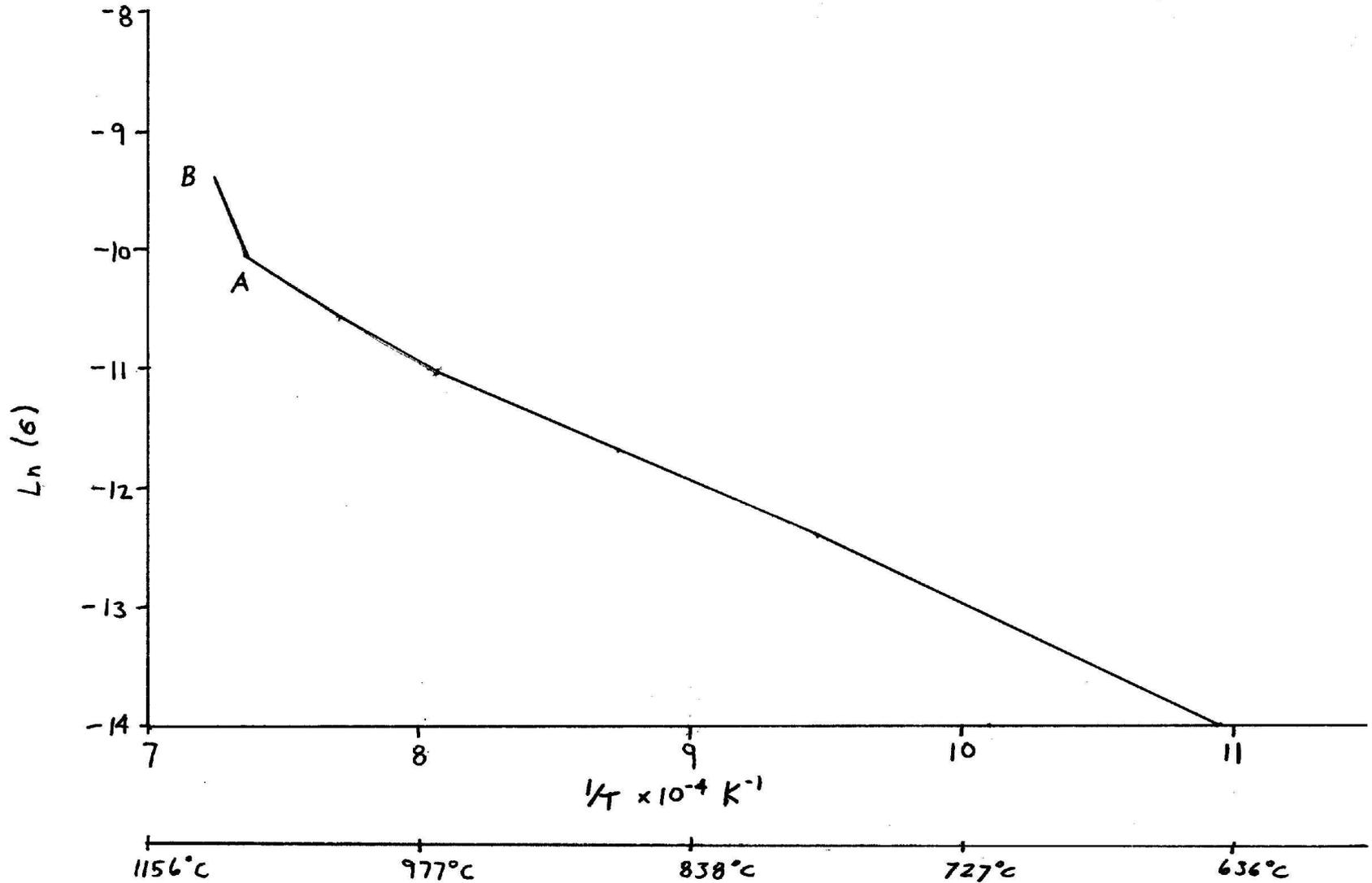
1.5 Hrs. at $\sim 1075^{\circ}\text{C}$

Optimally (?) Sintered

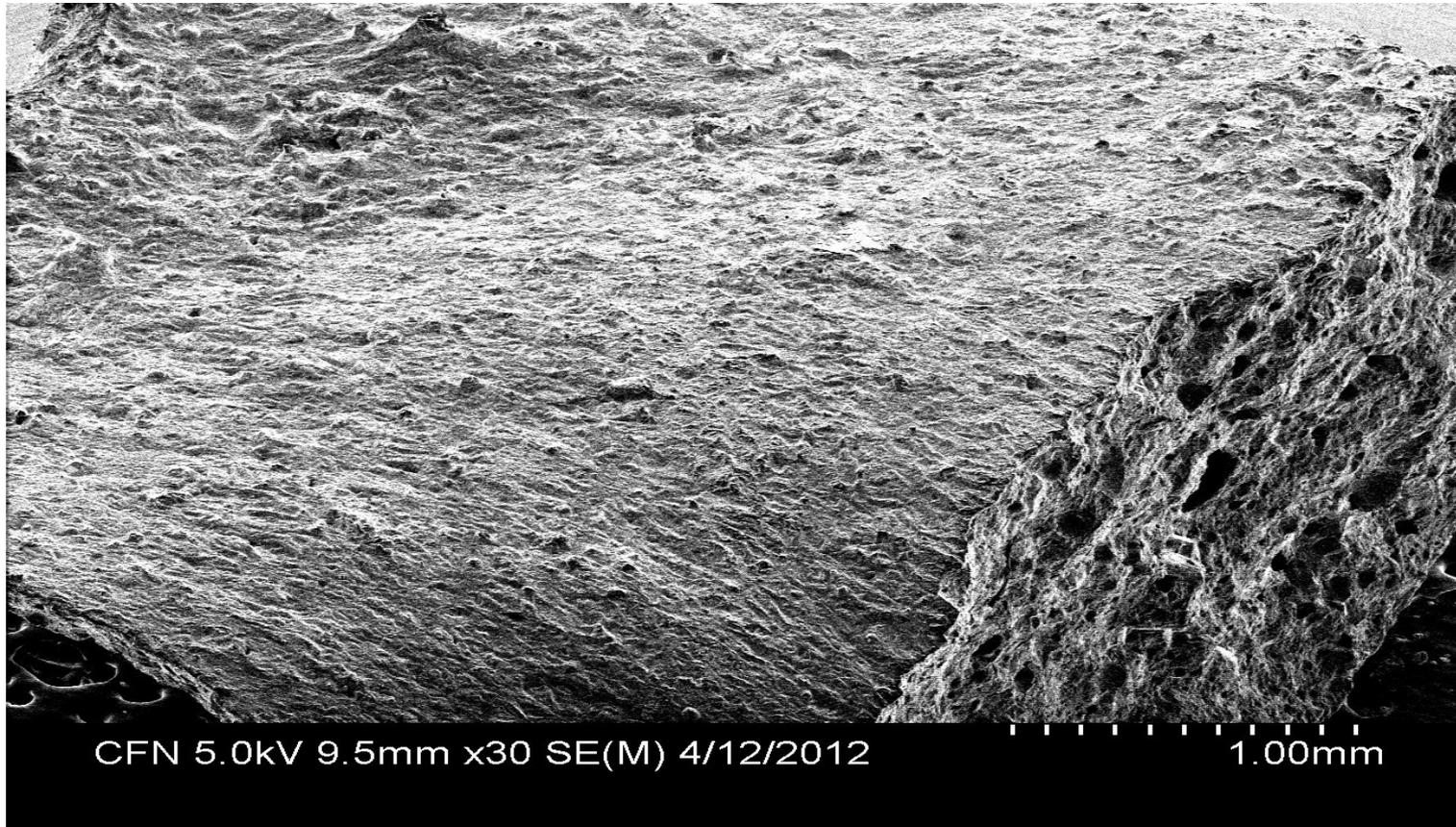


1.0 Hrs at
~ 1100°C

Optimally (?) Sintered

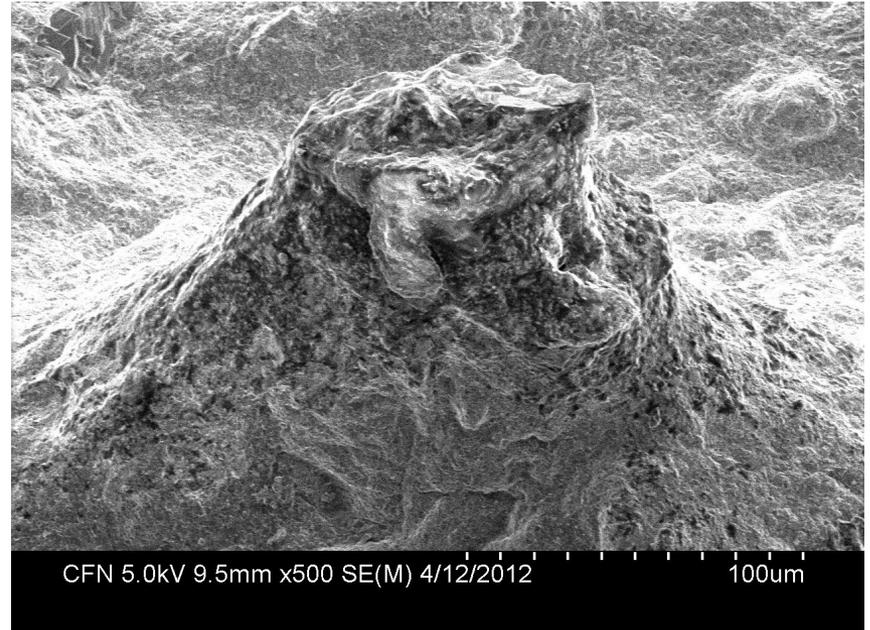
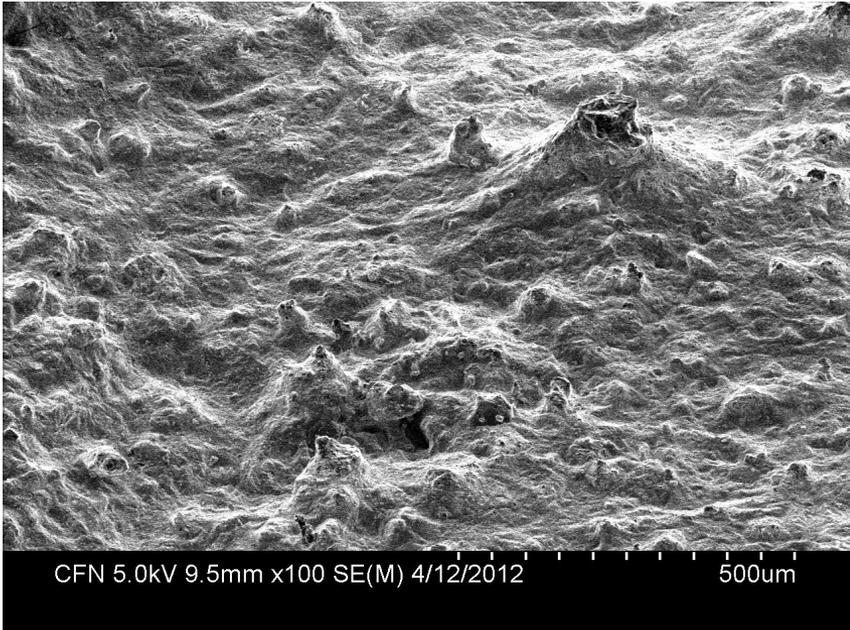


Completely Sintered (Melted)

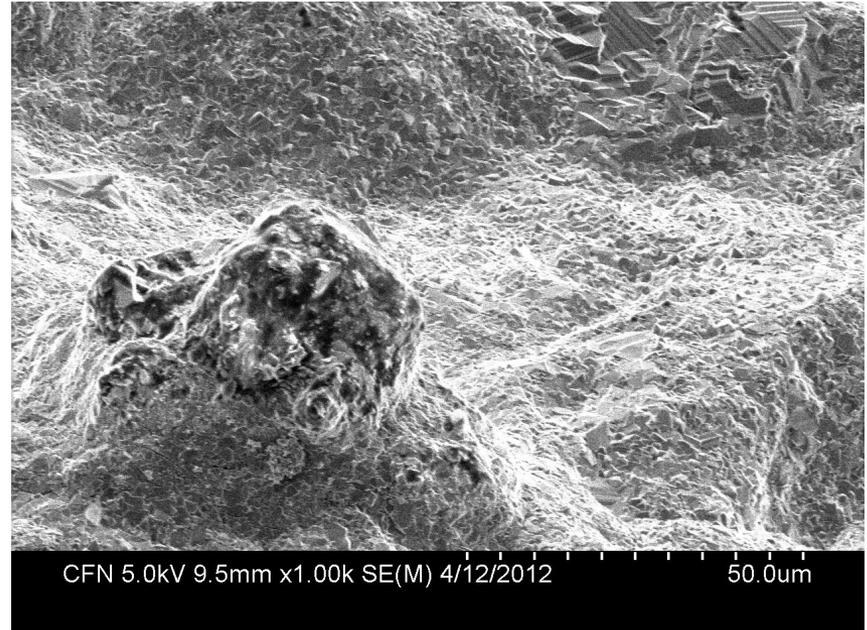
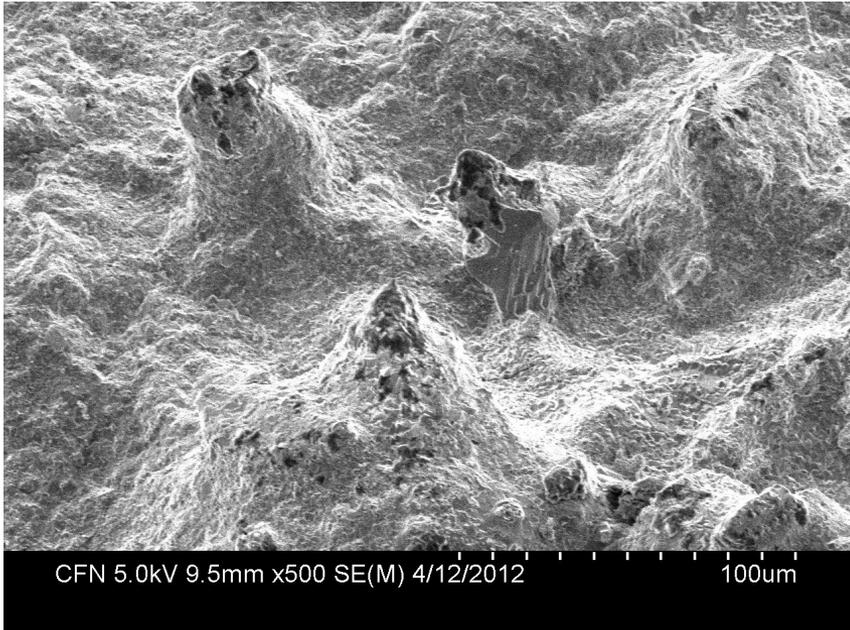


1.5 Hrs. at ~ 1160°C

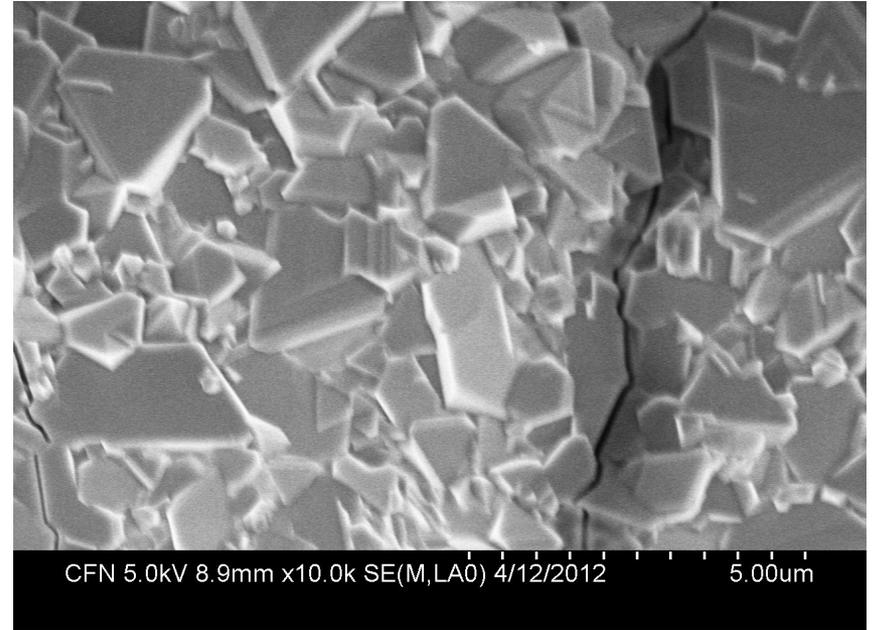
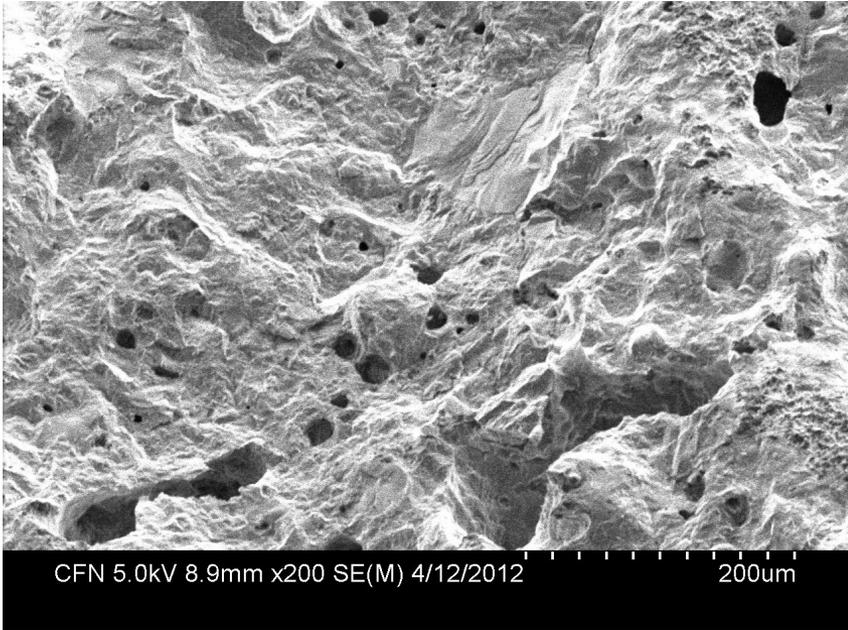
Completely Sintered (Melted)



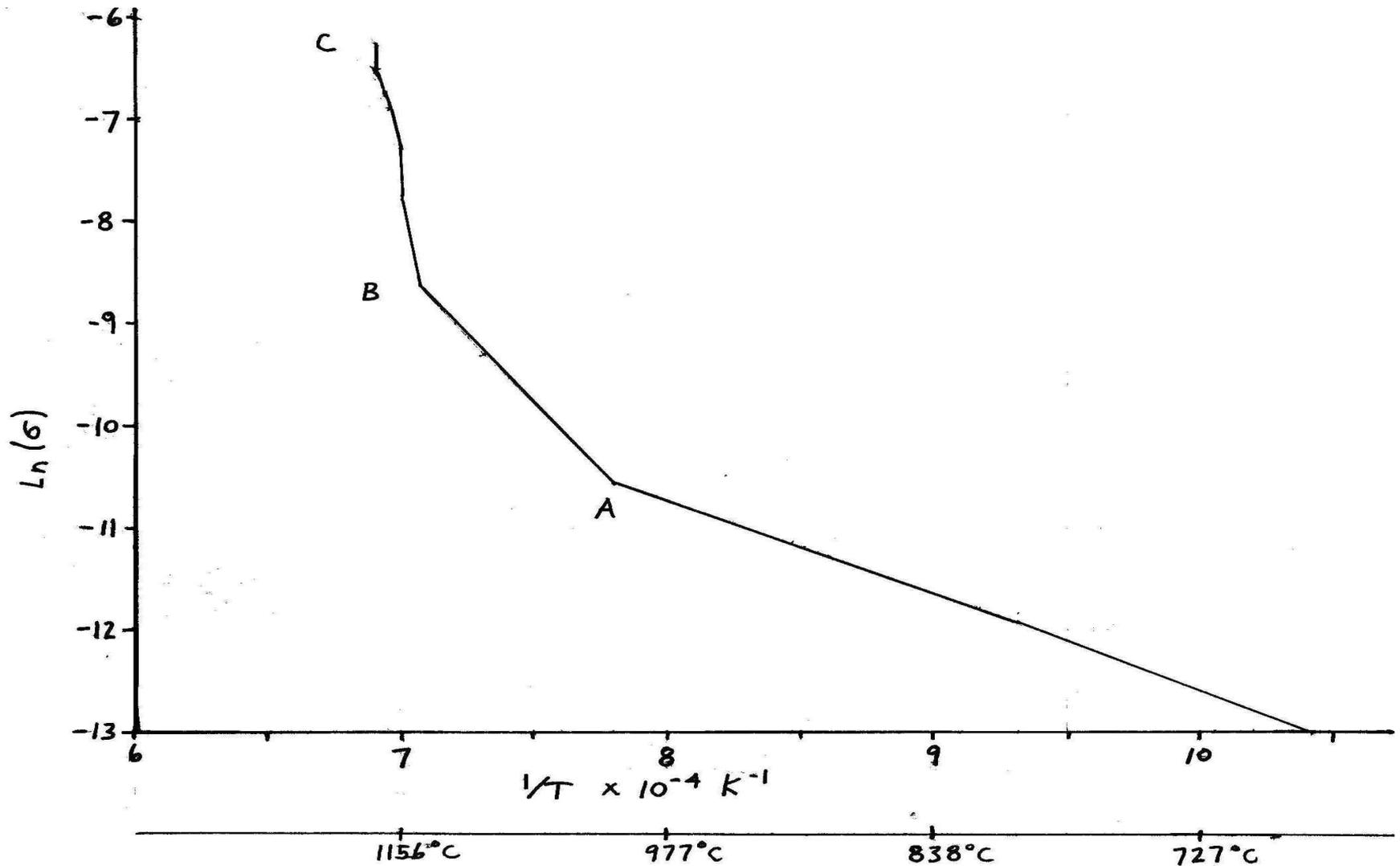
Completely Sintered (Melted)



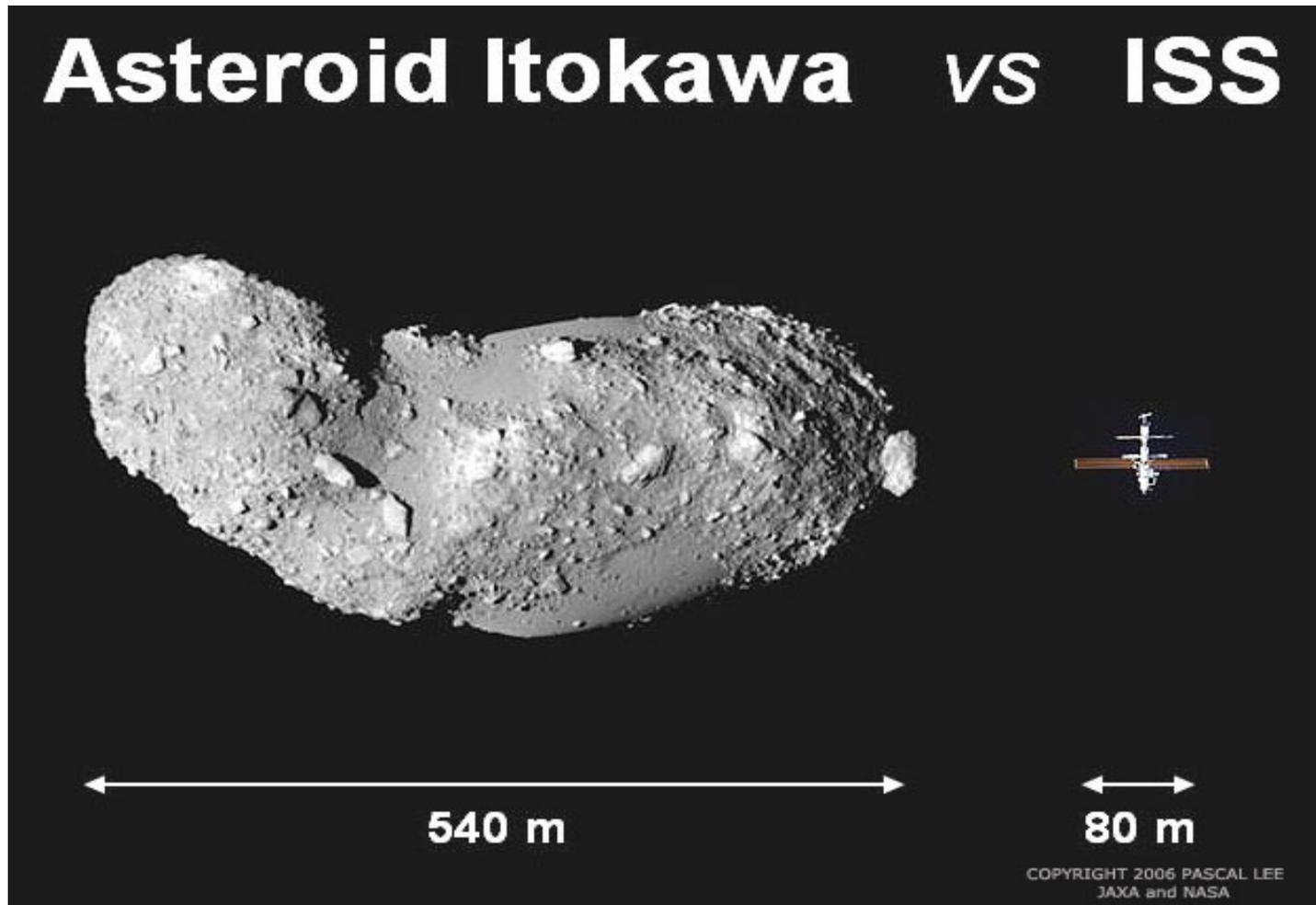
Completely Sintered (Melted)



Arrhenius Plot: 1.5 Hrs. at ~ 1160°C

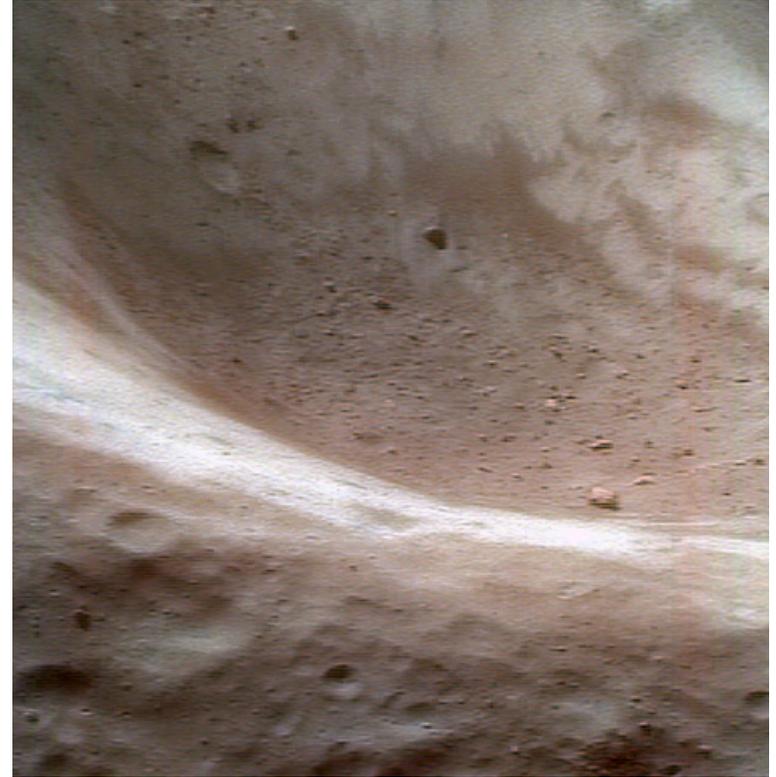


Asteroid 25143 Itokawa



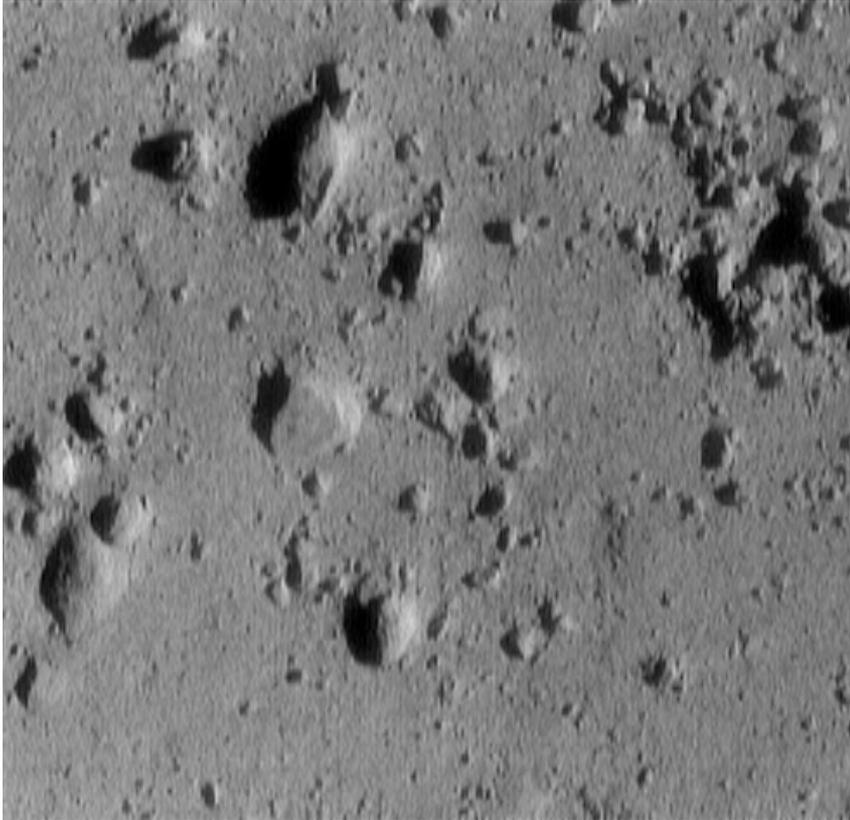
S-type Stony Asteroid

NEAR Asteroid Mission to 433 Eros

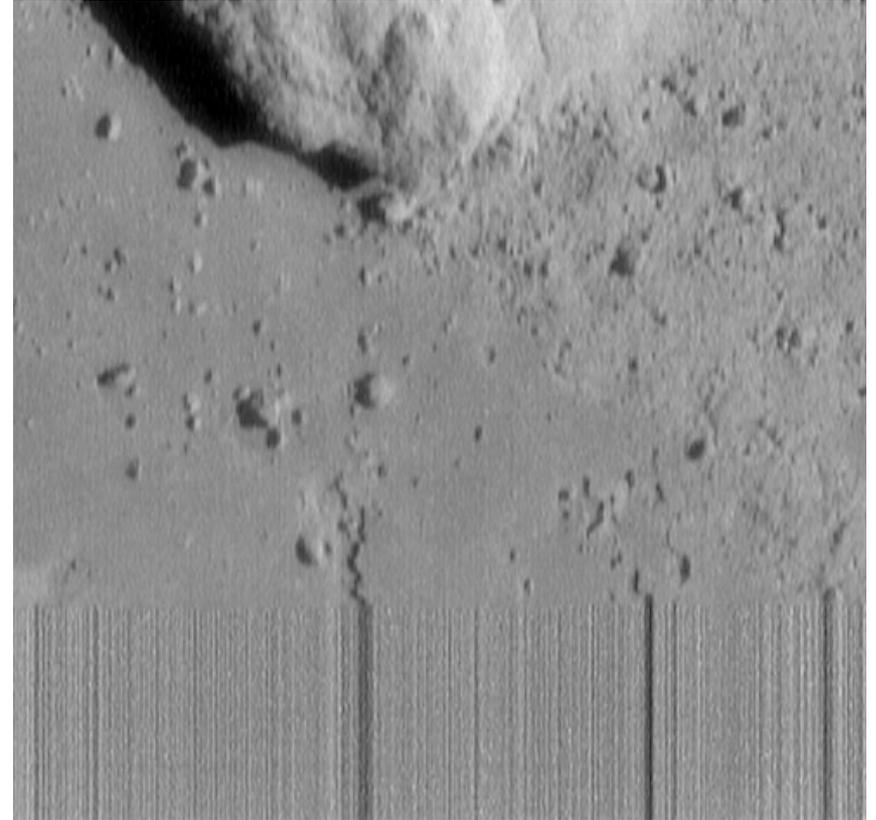


S-type Stony Asteroid

NEAR Asteroid Mission to 433 Eros

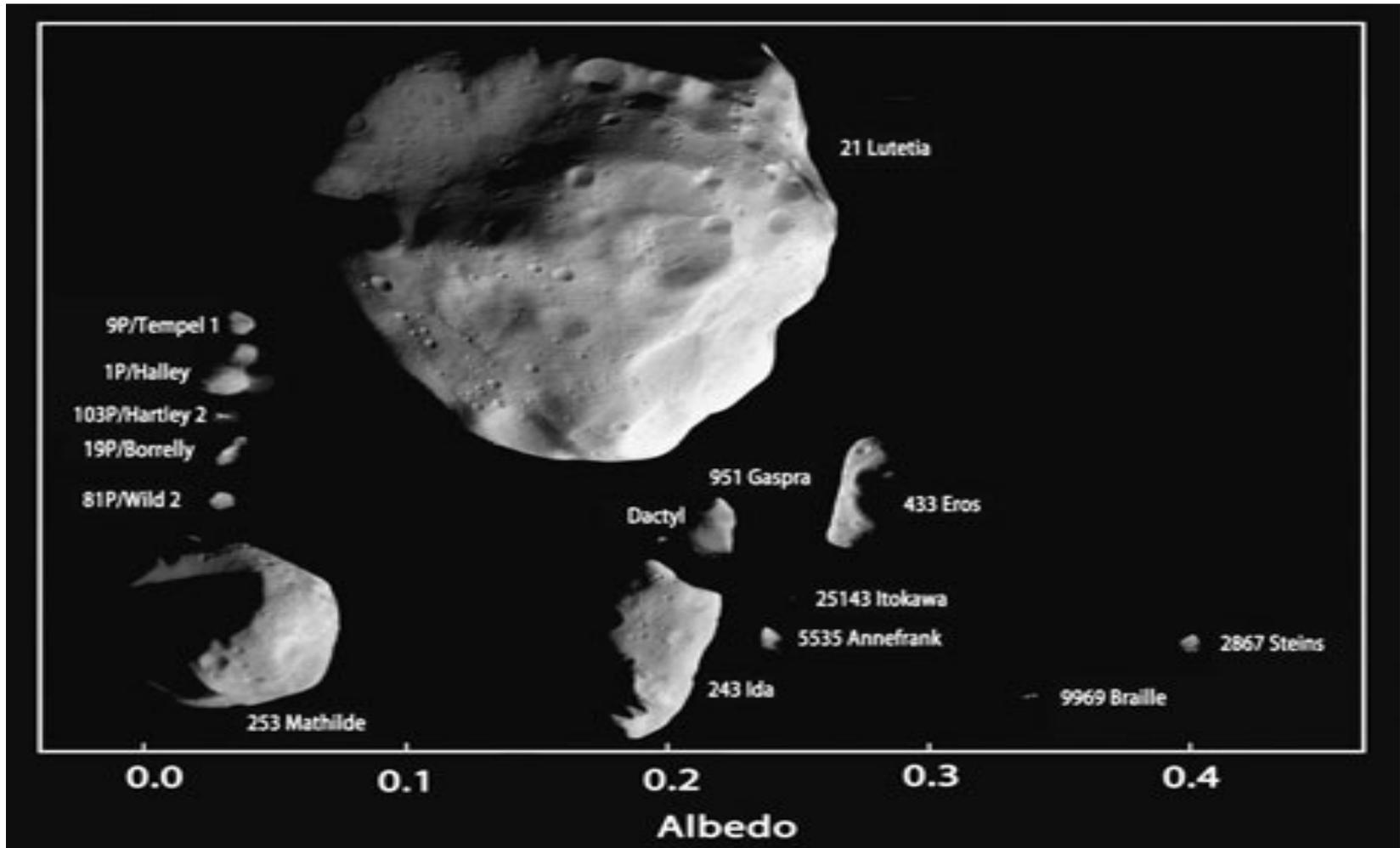


250 m



120 m

Asteroids and Comets Visited by Spacecraft (so far)



Similarities and Differences between Lunar Soil and Asteroid Regolith

Compositional Differences

Gravitational Environment-Induced Differences

1/6 G vs milli-G's

Micrometeorite Impacts

“Gardening” vs Sputtering

Similarities and Differences between Lunar Soil and Asteroid Regolith

Gravitational Environment-Induced Differences

+

Space Weather/Solar Wind Interaction

Moon: “Gardening” of Nanophase Iron

Asteroids: Reddening/Darkening of Surface (vener?)

Assist in Microwave Sintering and
Electromagnet-based Regolith Transport

Differences between Lunar and Asteroid Operations

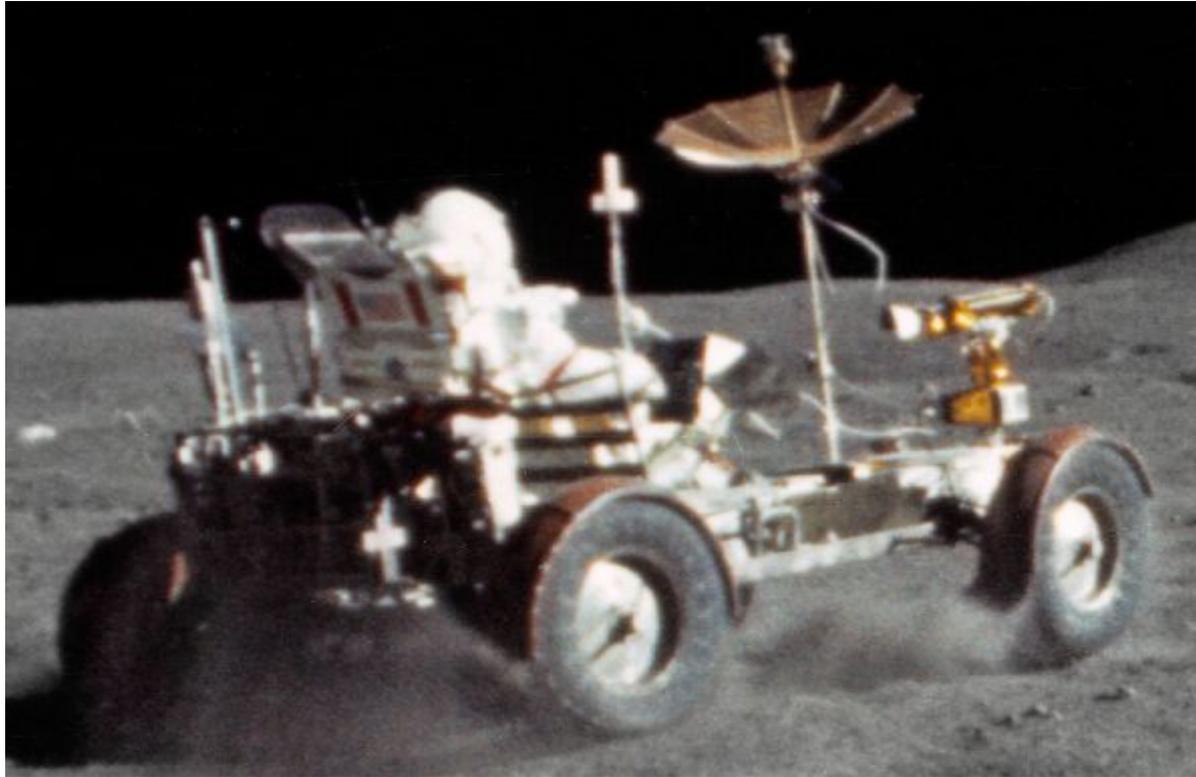
“Stand On” the Moon

VS

“Float Next To” an Asteroid

Raising Dust Cloud Fog by Asteroid Ops
Dust Cloud Mitigation: Electrostatic, Laser?
Slow Dispersion by Solar Photon Pressure

Differences between Lunar and Asteroid Operations



Don't try this on an Asteroid

Even Asteroids have Asteroids (Binary Asteroids)



NASA 2005

243 Ida (56 km) and Dactyl (1.4 km)

~ 16% of Asteroids are Binary

Even Asteroids have Asteroids (Binary Asteroids)

Name	Type	Diameter (km) (or dimensions)	Name of moon	Diameter of moon (km) (or dimensions)	Separation (km)
1862 Apollo	Apollo	1.7	S/2005 (1862) 1	0.08	3
3671 Dionysus	Amor	1.5	S/1997 (3671) 1	0.4	2.2
5381 Sekhmet	Aten	1	S/2003 (5381) 1	0.3	1.54 ± 0.12
7088 Ishtar	Amor	1.5?	S/2006 (7088) 1	?	?
(31345) 1998 PG	Amor	0.9	S/2001 (31345) 1	0.3	1.5
(35107) 1991 VH	Apollo	1.2	S/2001 (35107) 1	0.5	3.2
65803 Didymos	Amor	0.8	S/2003 (65803) 1	0.15 ± 0.05	1.1
(66063) 1998 RO ₁	Aten	0.9	S/2001 (66063) 1	0.36	0.8
(66391) 1999 KW ₄	Aten	1.2	S/2001 (66391) 1	> 0.36	2.6
69230 Hermes	Apollo	0.4	S/2003 (69230) 1	0.4	1
(85938) 1999 DJ ₄	Apollo	0.7	S/2004 (85938) 1	0.35	1.5
(88710) 2001 SL ₉	Apollo	1	S/2001 (88710) 1	0.31	1.8
(136617) 1994 CC	Apollo	0.7	? 2	both ~0.05	?
(137170) 1999 HF ₁	Aten	3.5	S/1999 (137170) 1	0.8	7.0
(162000) 1990 OS	Apollo	0.3	S/2003 (1990 OS) 1	0.045	0.6

Name	Type	Diameter (km) (or dimensions)	Name of moon	Diameter of moon (km) (or dimensions)	Separation (km)
(164121) 2003 YT ₁	Apollo	1	S/2004 (2003 YT ₁) 1	0.18	~2.7
(175706) 1996 FG ₃	Apollo	1.4	S/2001 (1996 FG ₃) 1	0.43	2.4
(185851) 2000 DP ₁₀₇	Apollo	0.80 (± 0.16)	S/2000 (2000 DP ₁₀₇) 1	0.30 (± 0.15)	2.622 ± 0.162
1994 AW ₁	Amor	0.9	S/2001 (1994 AW ₁) 1	0.5	2.1
1994 XD	Apollo	1?	S/2005 (1994 XD) 1	?	?
1998 ST ₂₇	Aten	0.8	S/2002 (1998 ST ₂₇) 1	0.12	4.5 ± 0.5
2000 UG ₁₁	Apollo	0.23 ± 0.06	S/2001 (2000 UG ₁₁) 1	0.10	0.337 ± 0.013
2002 BM ₂₆	Amor	0.6	S/2002 (2002 BM ₂₆) 1	0.1	1.5
2002 CE ₂₆	Apollo	3	S/2004 (2002 CE ₂₆) 1	0.2	5
2002 KK ₈	Amor	0.5	S/2003 (2002 KK ₈) 1	0.1	?
2003 SS ₈₄	Apollo	0.12	S/2004 (2003 SS ₈₄) 1	0.06	0.3?
2004 DC	Apollo	0.3	S/2006 (2004 DC) 1	?	?
2005 AB	Amor	1.2?	S/2005 (2005 AB) 1	0.3	2.5?
2005 NB ₇	Apollo	0.5 ± 0.1	S/2008 (2005 NB ₇) 1	0.2 ± 0.1	≥ 0.6
2006 GY ₂	Apollo	0.45	S/2006 (2006 GY ₂) 1	?	?

Binary Near Earth Asteroids

Asteroid Acquisition

OSIRIS-Rex (2016)

Asteroid Capture and Return (ACR) Mission

Meteorites

Asteroid – Meteorite Associations

Some Meteorites have the same
Reflectance Spectra as the
Parent (?) Asteroid

H E D ← Vesta

Howardite, Eucrite, Diogenite
family

Asteroid Composition Classification

C – Carbonaceous Chondritic ~75%

S – Stony Iron ~15%

M – Metallic

U - Unclassified

Meteorite Preparation

Remove Fusion Crust

Crush

Sieve

Test

Future Work

Repeat Tests Under High or Ultra High Vacuum

using JSC-1A and other Simulants:

JSC-1AF; MLS-1; FJS-1, 2, 3; OB-1;

NU-LHT-1M, 2M; Chenobi; etc.

Inclusion of Agglutinates and Nanophase Iron

Test Representative Meteorites

for Sinter-ability

Conclusions

Impedance Monitoring is a
Simple and Effective Means for
Monitoring the Onset of Grain Boundary Sintering
for Lunar Soil simulant JSC-1A

Acknowledgments

The author is grateful to the
Center for Functional Nanomaterials
Brookhaven National Laboratory
for the use of the
Hitachi 4800 Scanning Electron Microscope
under DOE contract
DE-AC02-98CH10886

Can the Lessons Learned in the
Sinter Monitoring and Control of
Lunar Soil Simulants be applied to
Near Earth Objects?

You Bet Your

Asteroid

It Can!