Synthesis of Zeolites on Board the Space Shuttle & the ISS with Spinoff Applications on Earth

Yeditepe

An Overview

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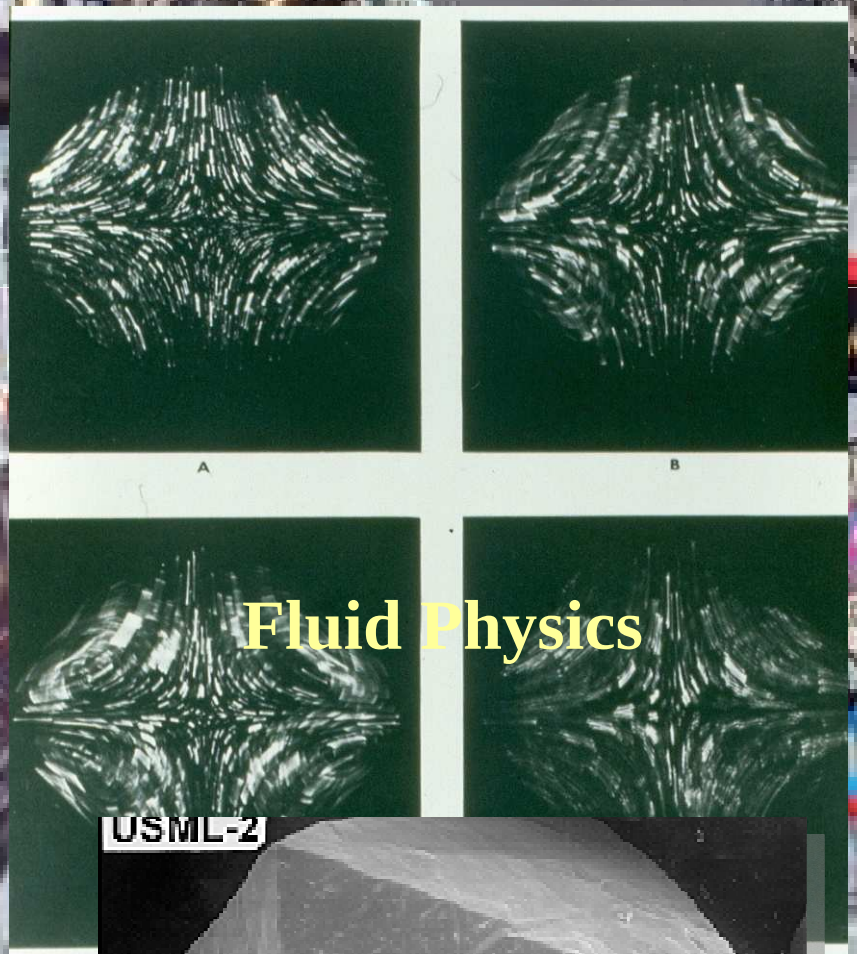
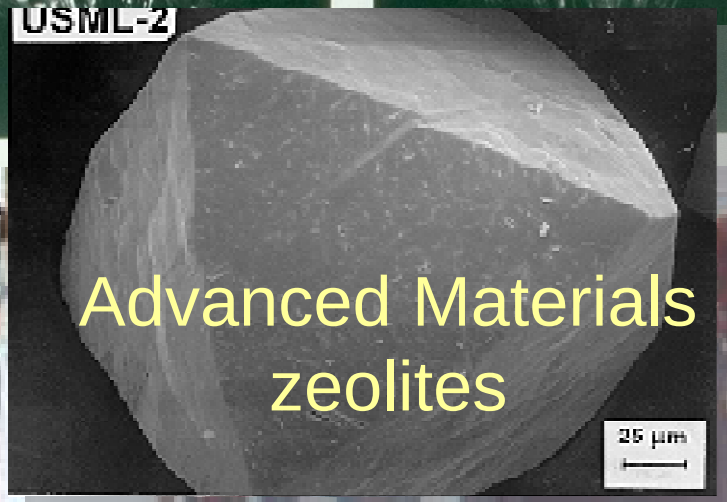
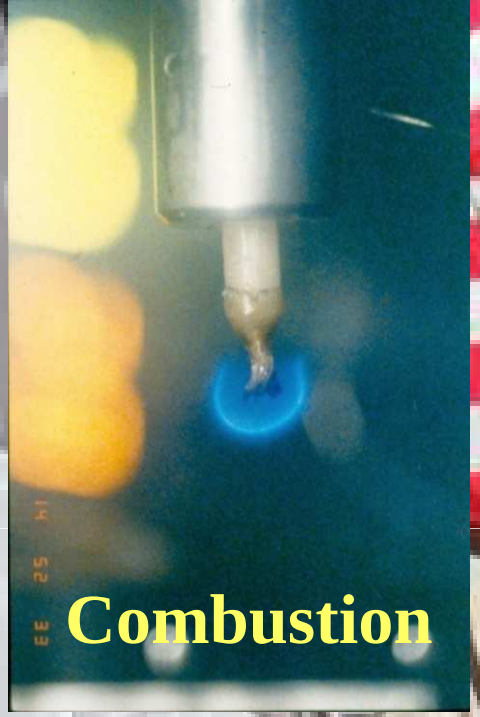
Experiments in Space



Fluid Physics





Combustion

Biomaterials, Plant growth

Human Life Sciences

Prof. N. Bac & Prof. A. Sacco‘s
ZCG (Zeolite Crystal Growth)
 Experiments in Space

Space Shuttle : STS-50, 57, 73, 107(1989-2003)

International Space Station :Increments 3,4,5,6 (2000-2003)

Why Grow Zeolites (Advanced Materials) Crystals in Space?

SCIENTIFIC RATIONAL

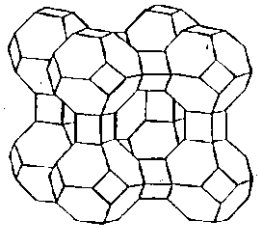
- Eliminate sedimentation
- Diffusion limited growth
- Secondary nucleation effects minimized

Overall effect : crystals with fewer defects

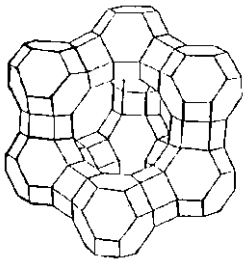
Economic Rational (Zeolites)

- Chemical Process Industry's major catalytic material,
- Wide range of applications,
- Exotic use

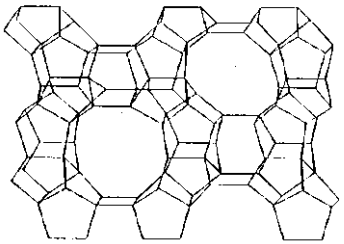
ZEOLITE- MOLECULAR SIEVES



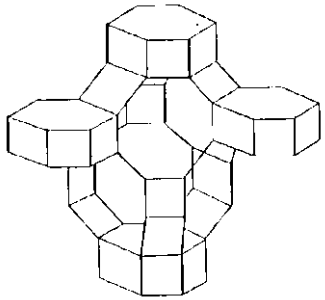
ZEOLITE A



FAUJASITE



PENTASIL LAYER



CHABAZITE

Zeolites (more than 50 types)

Alumino silicate crystals

$$M_{2/n}\text{-}Al_2O_3\text{-}xSiO_2\text{-}yH_2O$$

Nanoporous crystal structure.

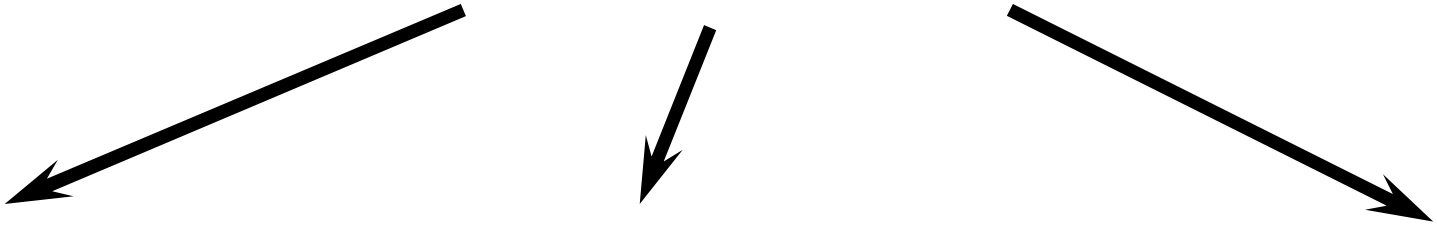
Some areas of use: - Economic and social impact

- Cat. cracking in petroleum refineries (gasoline production)
- ion exchangers (purify water , additive in powdered detergents)
- nanocomposites , antimicrobials,

hosts for microencapsulation.

Worldwide market over 2.5 billion USD

Categories of Crystals Growth in Space



SOLUTION

Proteins

(Drug Design)

Zeolites

(petrochemicals

Fuel cells,

Antibacterial agents)

VAPOR Phase

Mercury Cadmium Telluride

$Hg_{1-x}Cd_xTe$

Electronics

From MELTS

Gallium Arsenide

GaAs (selenium doped)

Mercury Zinc Telluride

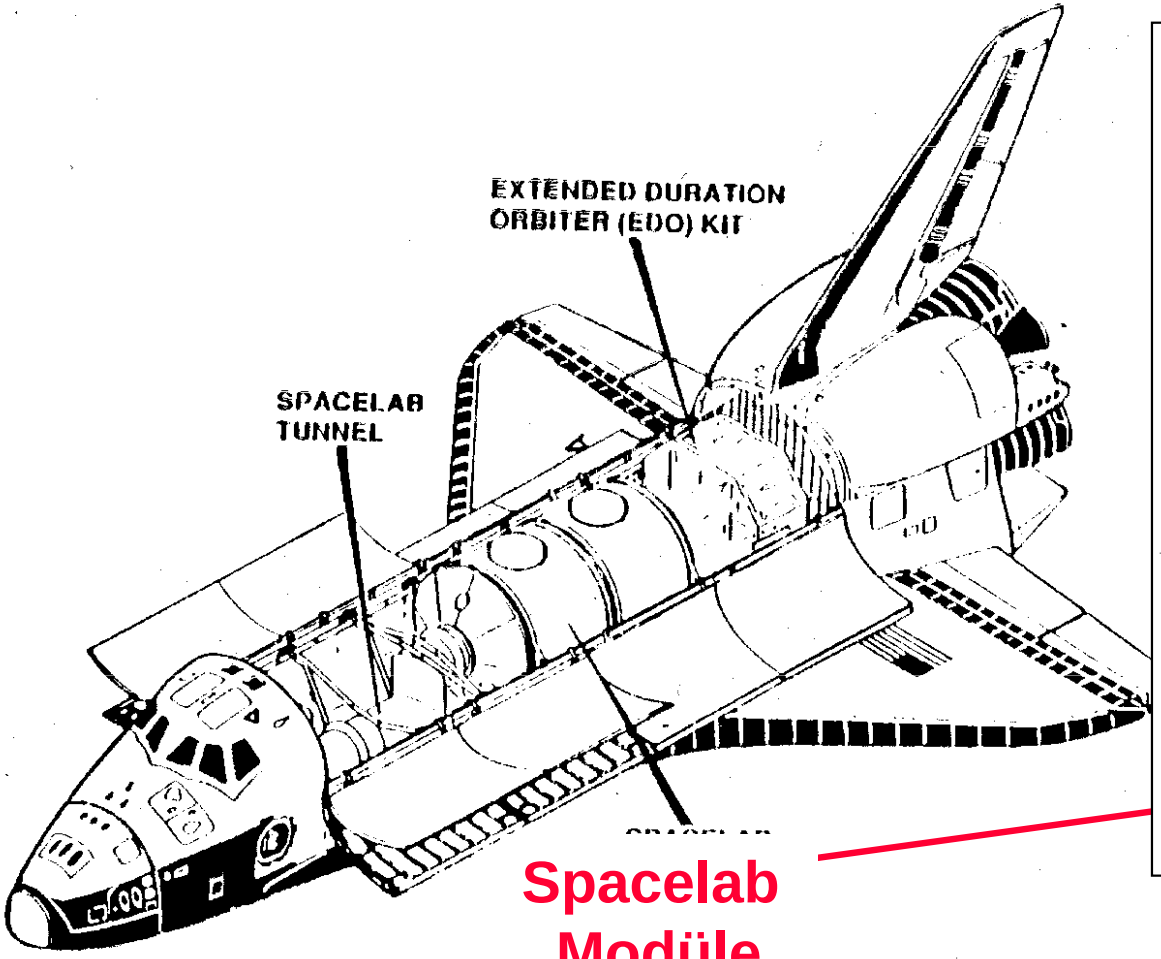
$Hg_{1-x}Zn_xTe$

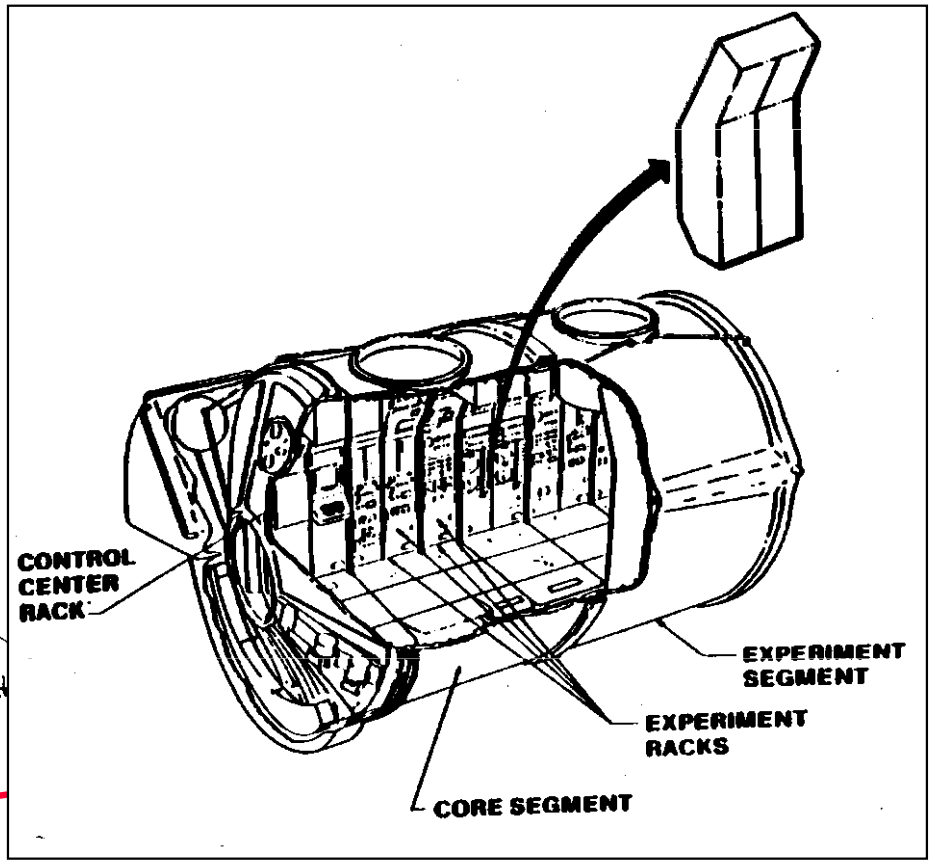
Cadmium Zinc Telluride

$Cd_{1-x}Zn_xTe$

Electronics

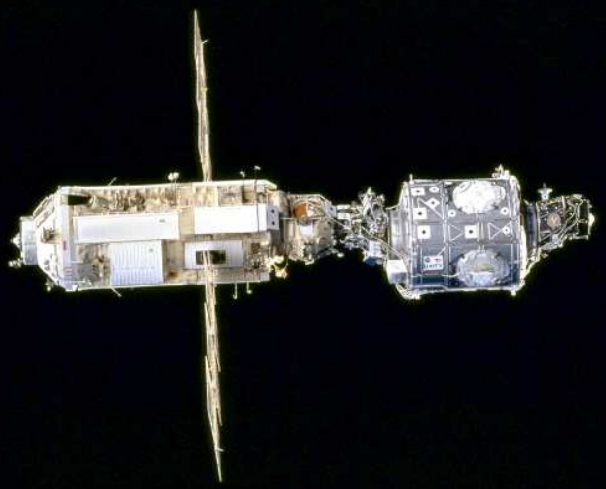
Where Were These Experiments Held ?
Columbia Space Shuttle STS-73
USML-2 (United States Microgravity Lab- 2)



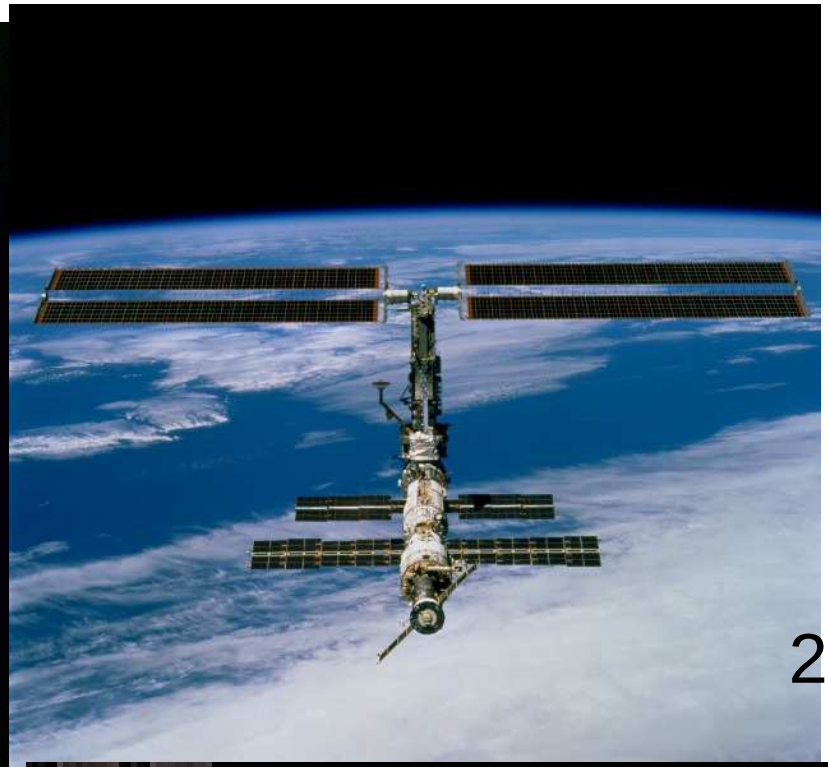


Spacelab Modüle

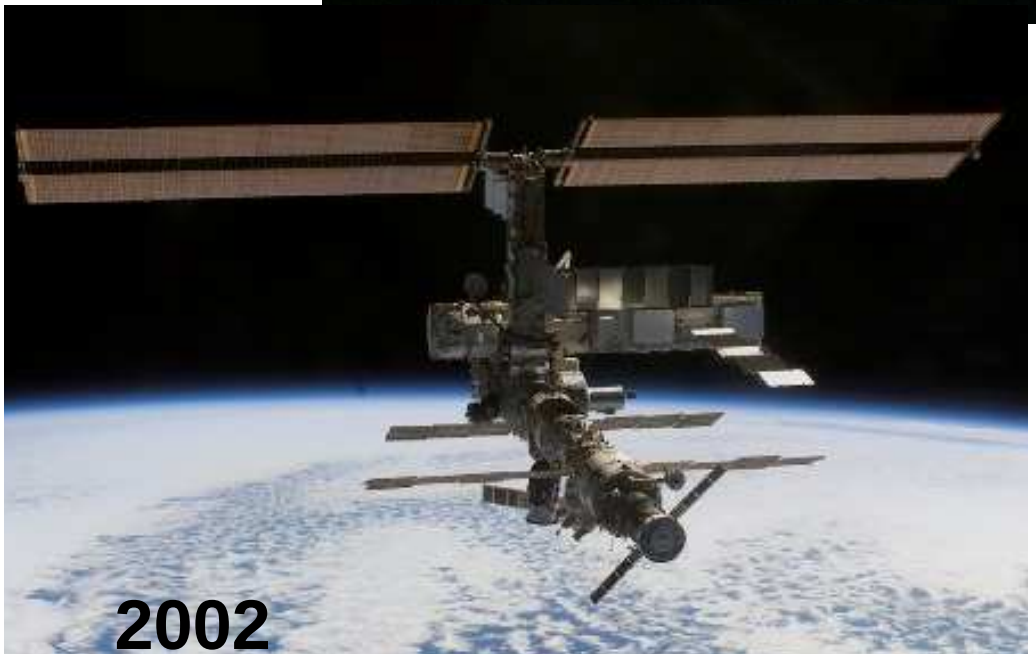
ISS ASSEMBLY



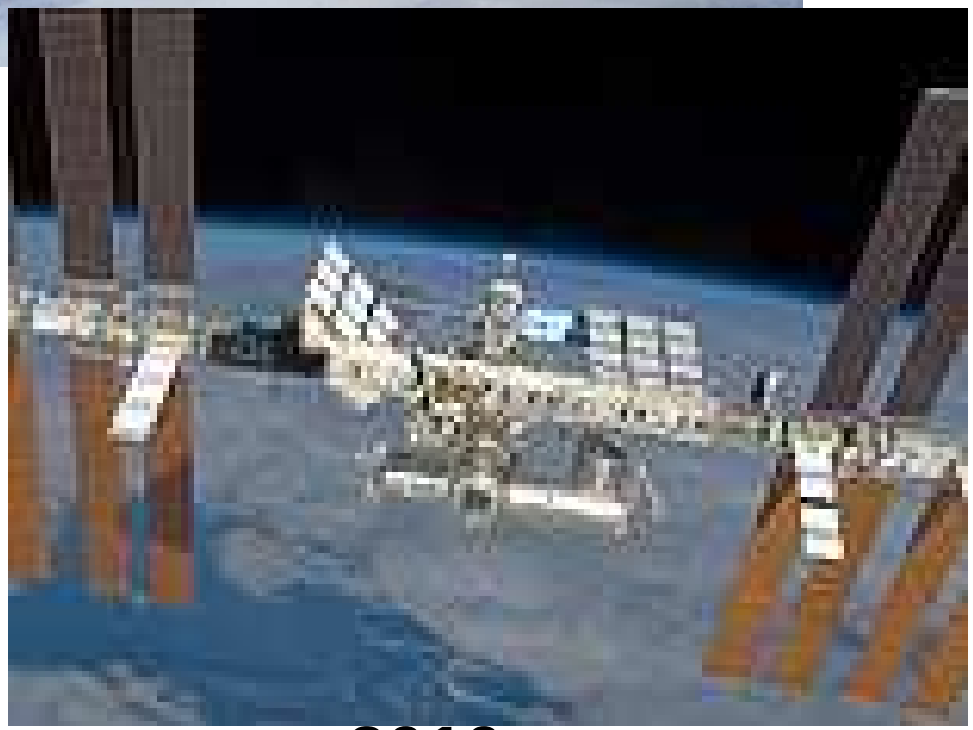
1998



2000

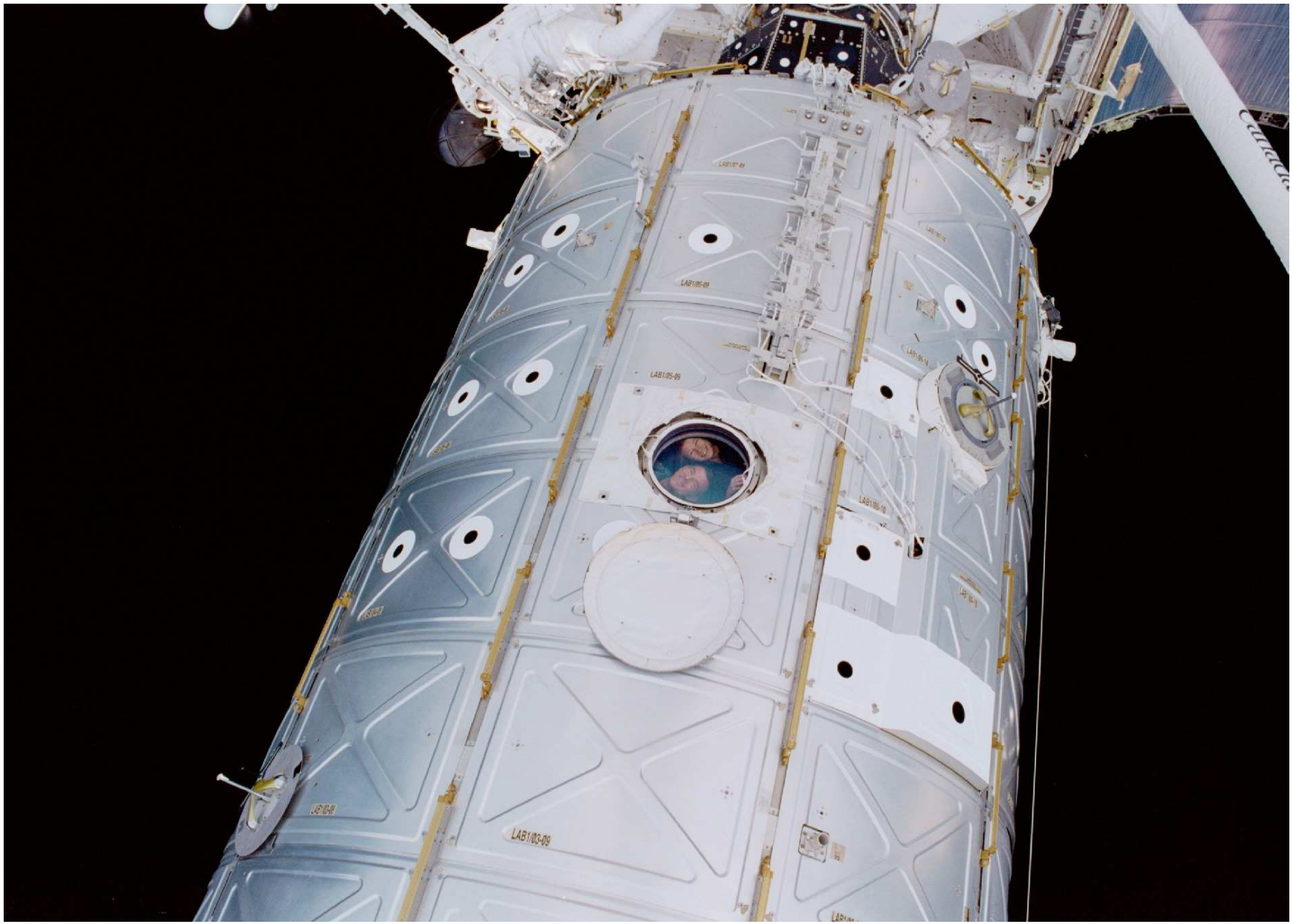


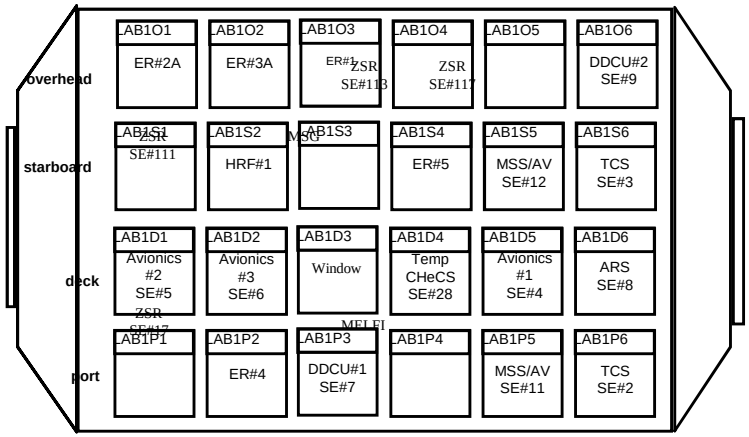
2002



2010

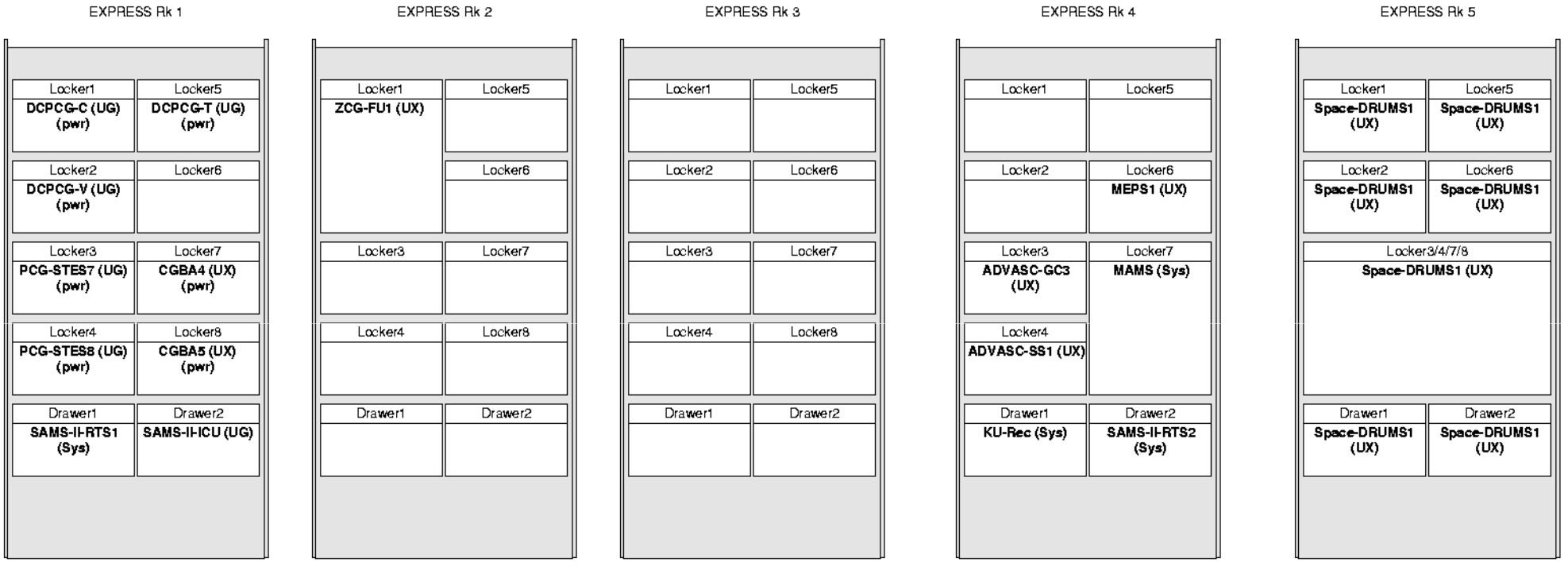
ISS - US Lab (Destiny)





International Space Station - US Lab

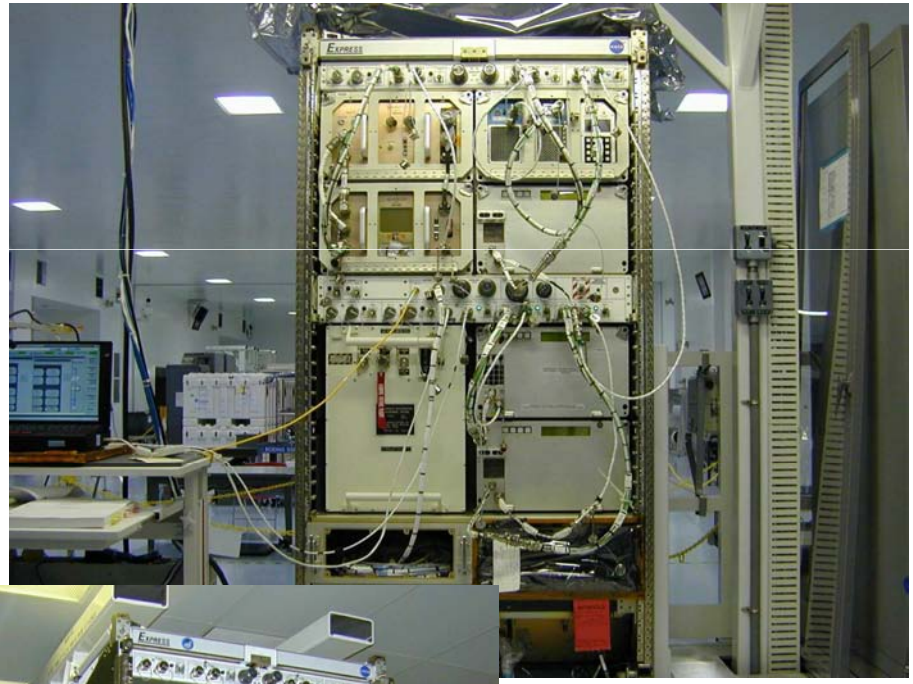
EXPRESS Rack

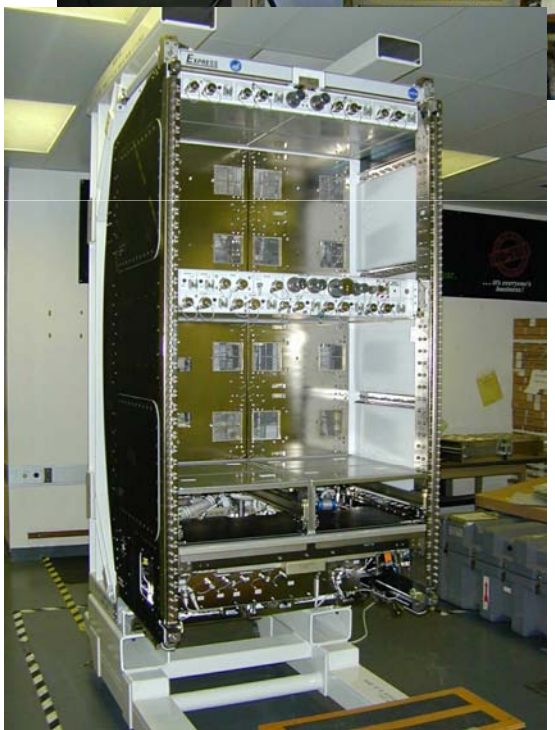


EXpedite **PR**ocessing **E**xperiments on **S**pace **S**tation

Facilities

“Expedite the Processing of Experiments to Space Station Rack” (EXPRESS Rack)





FACILITY OBJECTIVE

Provides simple, standard interfaces to accommodate drawer-level, locker, and modular-type payloads.

The EXPRESS Rack concept provides the capability for a simple and shortened integration cycle.

FLIGHT OPERATIONS SUMMARY

Transported in MPLM to Orbit with partial subrack payload complement

Rack transferred to Destiny and installation checkout performed

SPD

"Zeolite Crystal Growth Furnace" (ZCG)

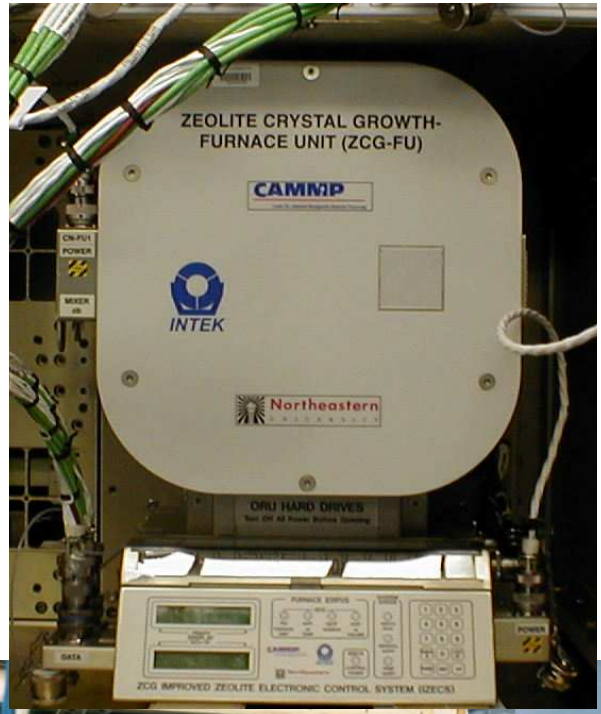
Al Sacco, Ph.D.; Nurcan Bac, Ph.D. Center for Advanced Microgravity Materials Processing (CAMMP), Boston

RESEARCH OBJECTIVE

- Use the ISS Microgravity environment to grow larger crystals with improved defect structure for zeolites, or other materials to enhance their adsorption properties and catalytic performance in important chemical processes, electronic device manufacture, and other applications

FLIGHT OPERATIONS SUMMARY

- ZCG is mostly autonomous except crew interaction required for:
  - Start up
  - Shutdown
  - Sample change out (experiment runs last 10-20 days)
  - Monitoring: Photography and check temperatures at predetermined intervals
  - Packaging samples for return to Earth



ZCG Furnace on its way to The International Space Station

Al Sacco Jr.

Nurcan Bac

Experiments Are Transported to ISS

MPLM at Kennedy Space Center (KSC , Florida)

MPLM is being transported to ISS

MPLM=Multi Purpose Logistics Module

On-Orbit Operations

Unstow sample autoclaves, and place in the furnace

Configure furnace, and IZECS for operation, Automix autoclaves, and start heat-up

During furnace heat-up and crystal growth limited monitoring takes place. The crystal growth process is unattended.
All heater tubes are computer controlled
Commands can be uplinked (smart furnace)
Temp and power, health and status data are downlinked every second

Furnace deactivation and power down.

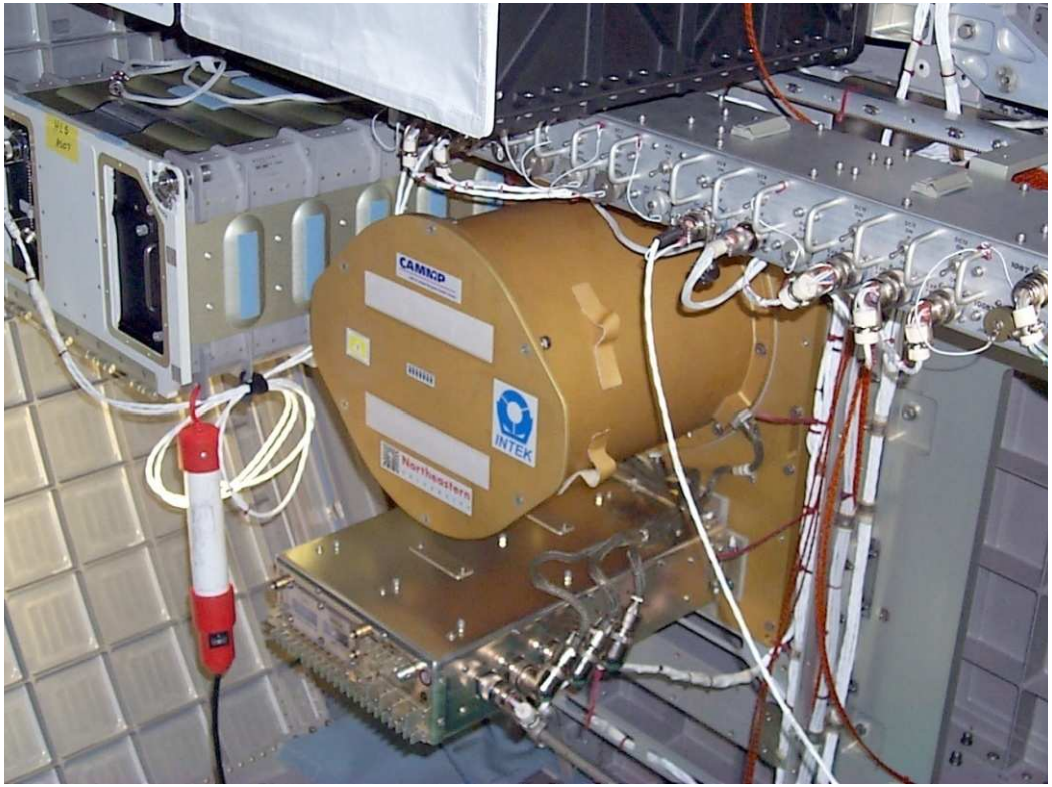
Unload and stow autoclaves.

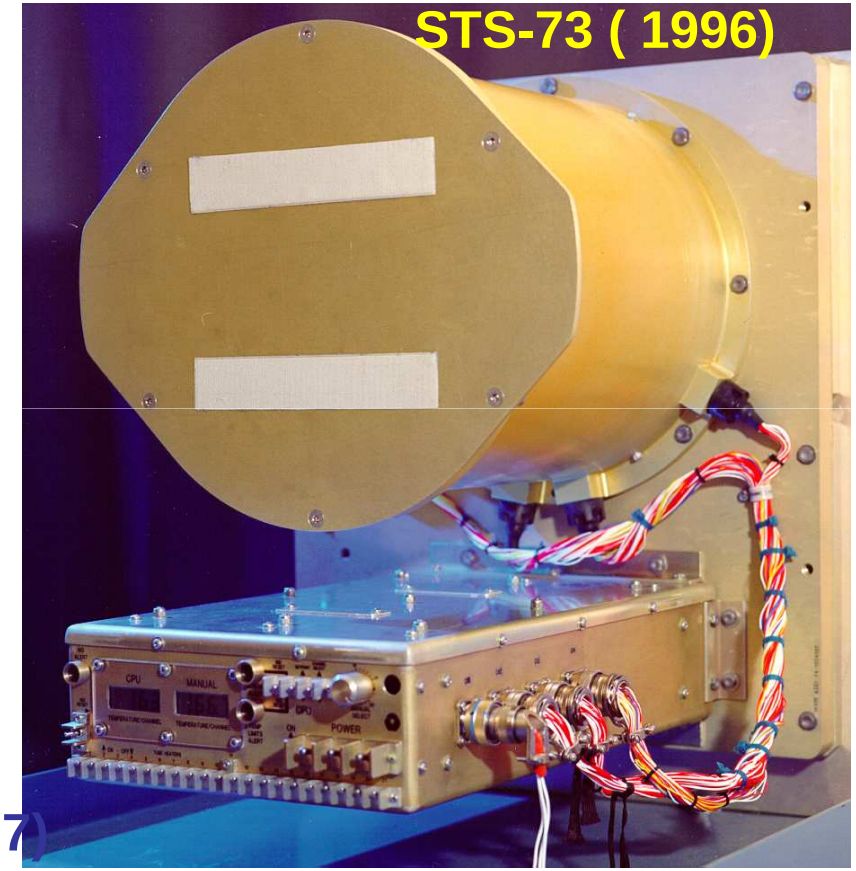
ZCG (Zeolite Crystal Growth) FURNACE ON THE SPACE SHUTTLE –

Al Sacco Jr. and N. Bac

Specs

- **Power (heat-up) :150 - 200 W**
- **Power (steady state) : 90 - 170 W**
- **Weight (loaded) : ~ 75 kg**
- **Temperature : 88 - 190 ° C.**
- **Samples : 38 autoclaves**





ZCG Furnace was lost with Columbia (STS-107) when it crashed during entry 2/1/2003

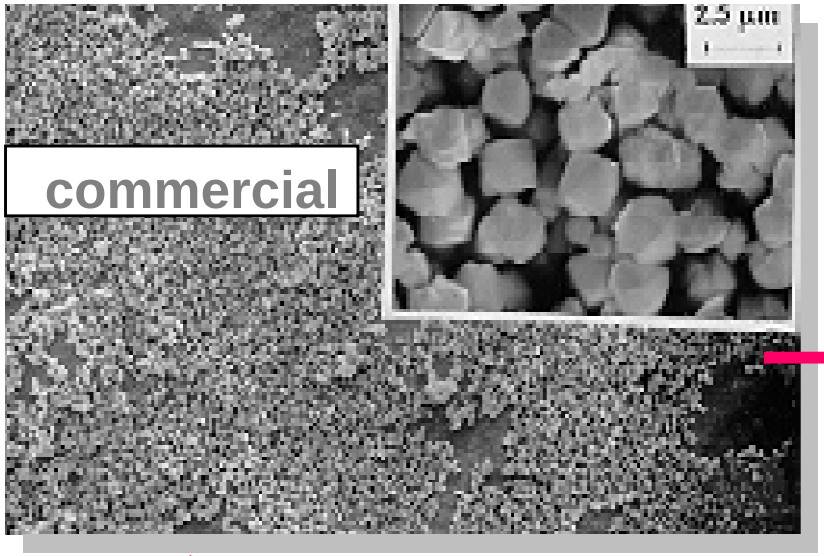
FLIGHT AUTOCLAVES

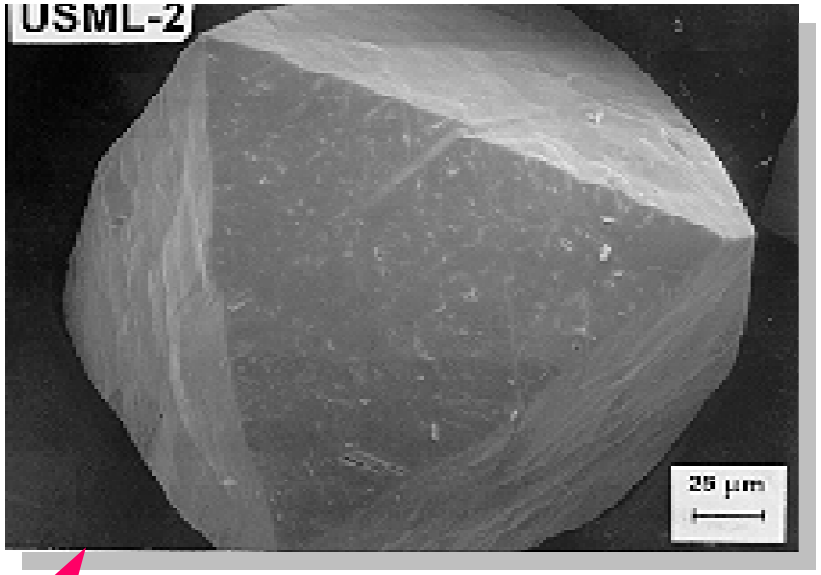

Autoclaves on the

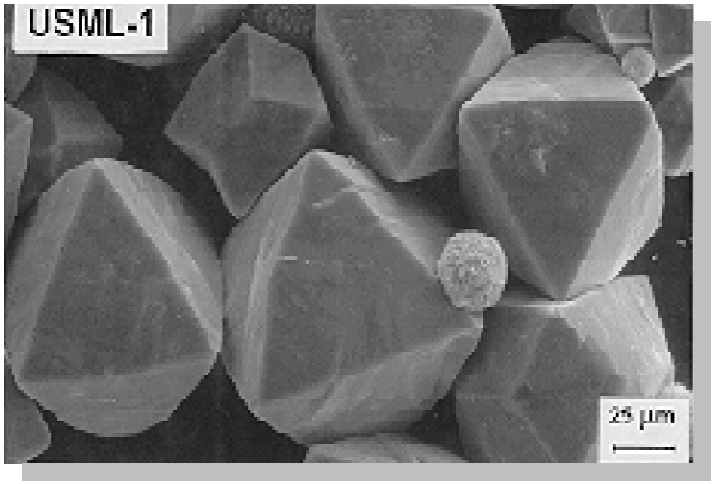
- Teflon lined aluminum or titanium autoclaves
- Automix design has a single autoclave coupled with a housing with a DC motor/mixer per furnace tube.

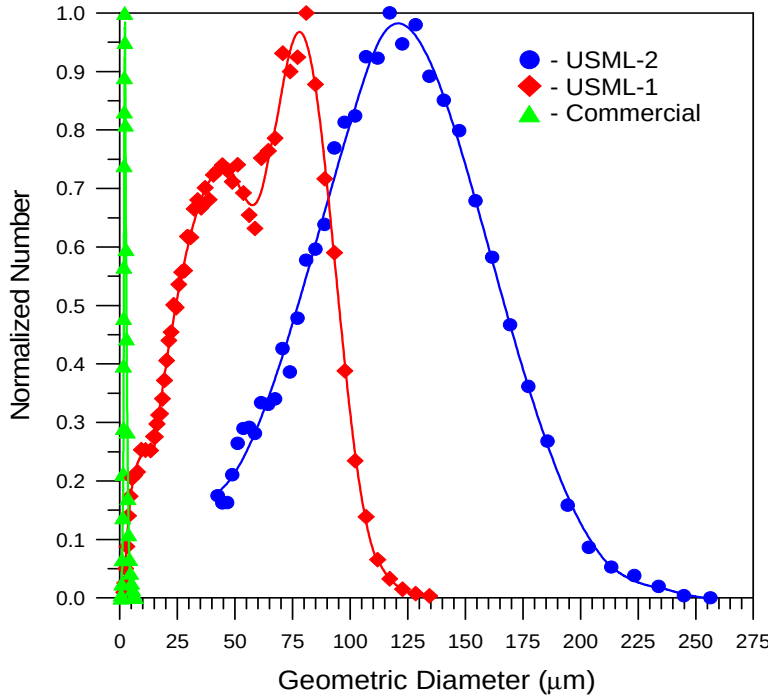

Autoclaves on ISS

Space Development - Zeolite X

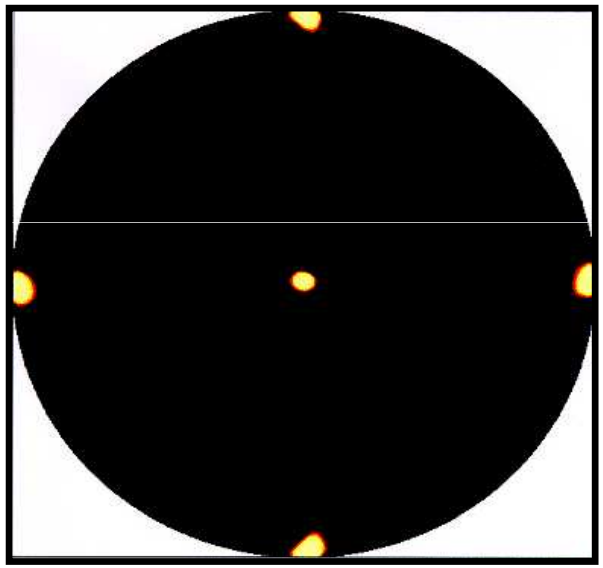


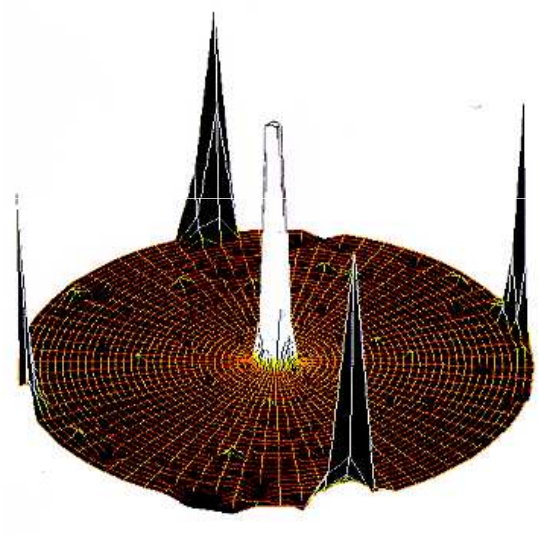




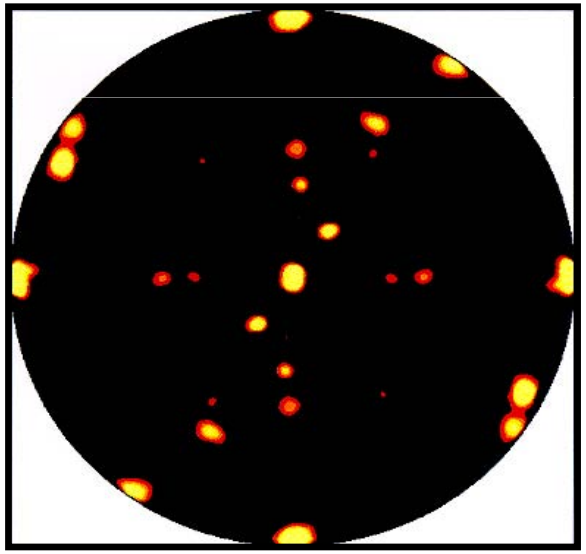


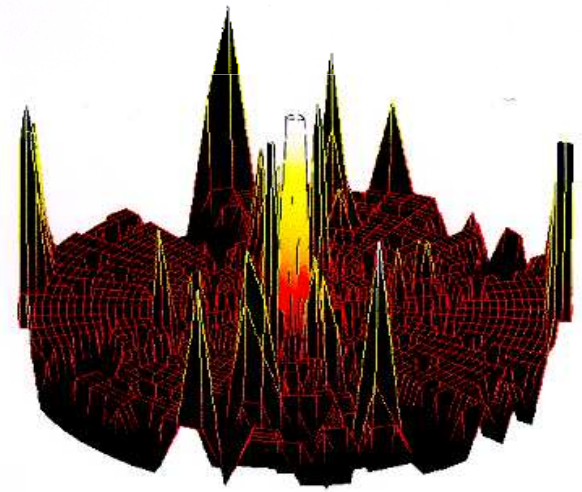
Single Crystal X-Ray - Zeolite A





SPACE

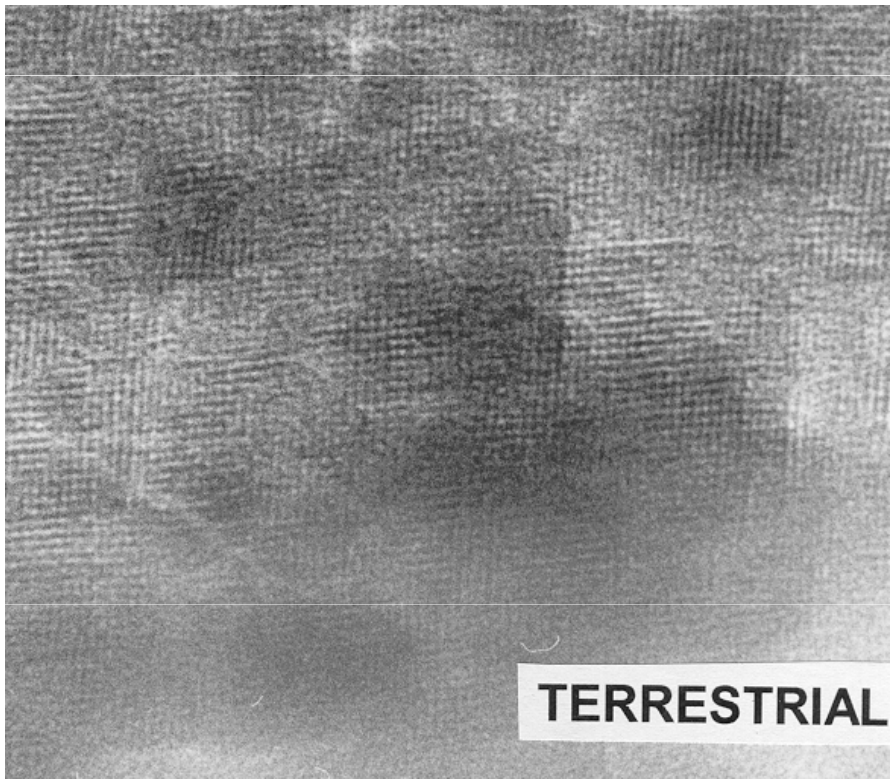




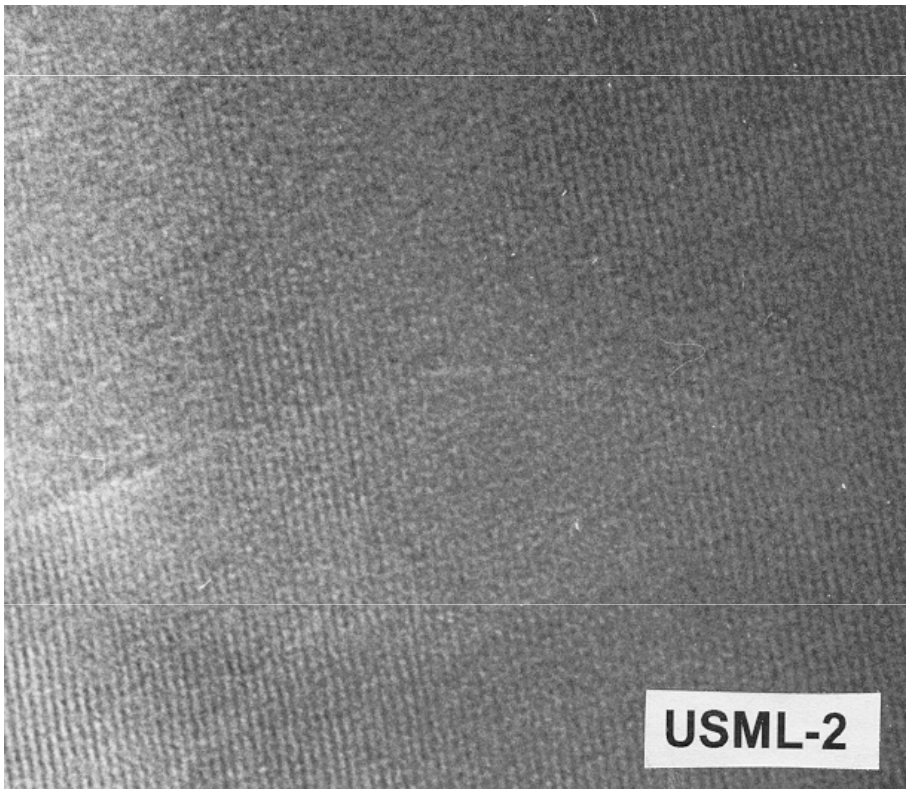
TERRESTRIAL

Transmission Electron Microscope (TEM)

Zeolite Beta- Defect-Free Crystal



Ground



Space

CONCLUSION –Space Results

STS-73 Columbia Crew

- ***Large and structurally defect-free zeolites*** *are grown in space.*

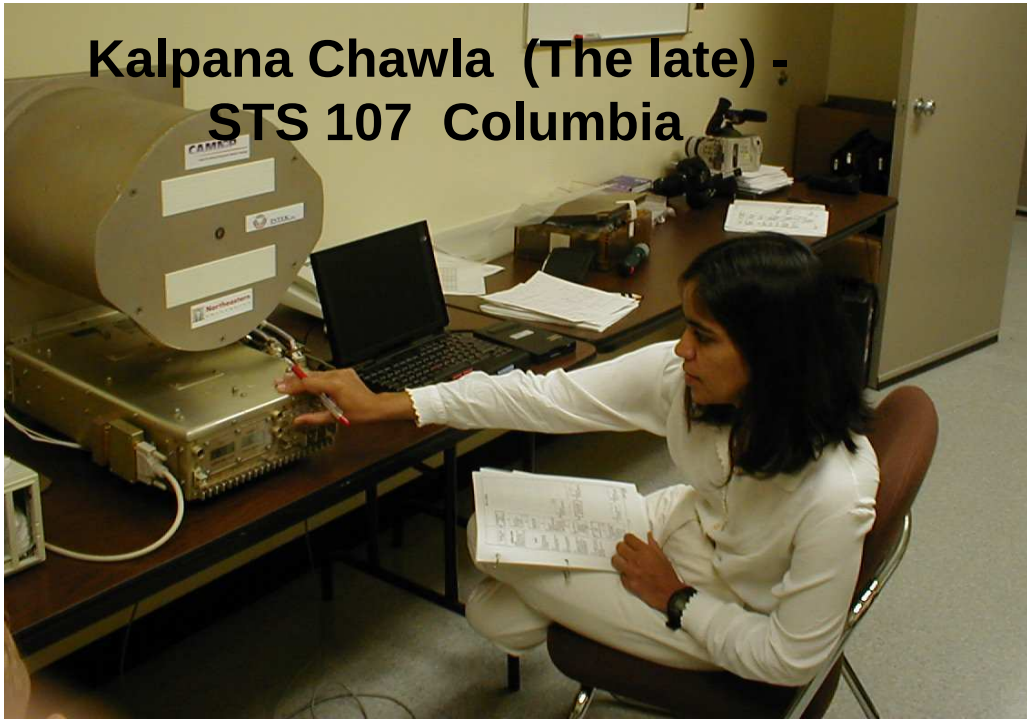
These become benchmark crystals.

- *AFM, results indicate smooth surface for space grown zeolite crystals with distinct growth planes.*
- *Knowledge base from space grown products enable us to synthesize them better on earth.*

ASTRONAUTS AND COSMONAUTS TRAINED ON THE ZCG EXPERIMENT

Bonnie Dunbar - STS-50 (1992)
Albert Sacco Jr. - STS-73 (1995)
Vladimir Dezhurov - ISS Inc. 3 (2000)
Yuri Oniferenko - ISS Inc. 4 (2001)
Carl Walz - ISS Inc. 4 (2001)
Peggy Whitson - ISS Inc. 5 (2001)
Ken Bowersox - ISS Inc. 6 (2002)

STS – Space Shuttle Columbia flight crew
ISS – International Space Station Crew
Inc = Increment # on orbit



Kalpana Chawla (The late) - STS 107 Columbia

Laurel Clark (The Late) - STS 107 Columbia

Spinoff applications – Zeolites in Health Sciences

- Known biological properties
- Long term stability
- Ability to reversibly bind to small molecules
- Size and shape selectivity
- Low cost

Auerbach et. al., "*Handbook of Zeolite Science and Technology*", New York: M. Dekker, 1–2, **2003**.

Silver Ion, Ag^+

- Antibacterial effect known since the ancient times
- Strong antibacterial activity
- High stability
- Very broad spectrum
- Exerts its effect through binding to bacterial DNA and inhibiting the most important metabolic activities of the cell such as transport processes and respiration.
- Metallic silver has only slight antibacterial effect when compared to Ag^+.

Antibacterial E-coli results

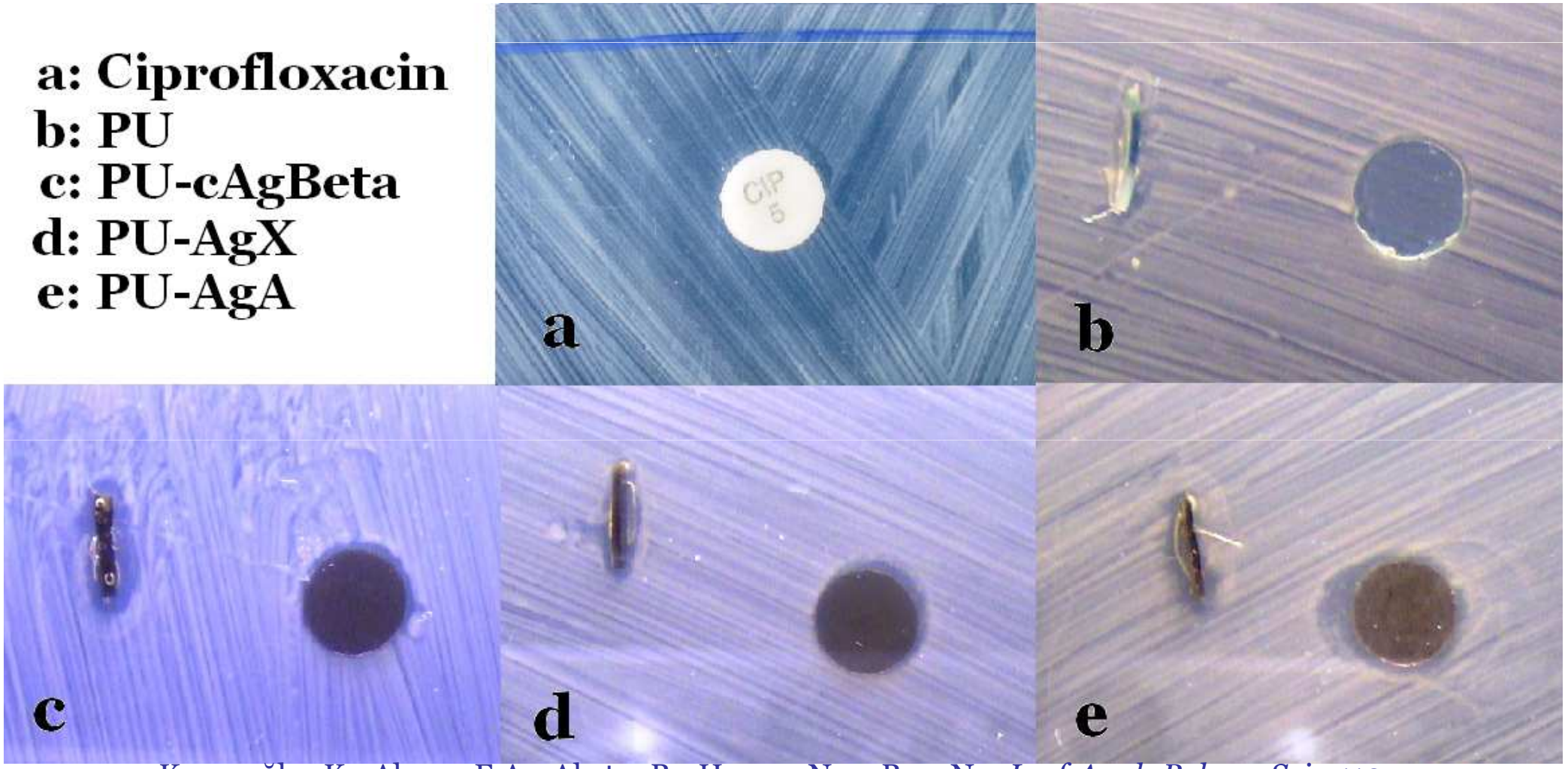
Effect Zone of Ag^{+}ion exchanged zeolite A

Ion Exchanged Sample

Effect Zone of Ag^{+}ion exchanged zeolite X

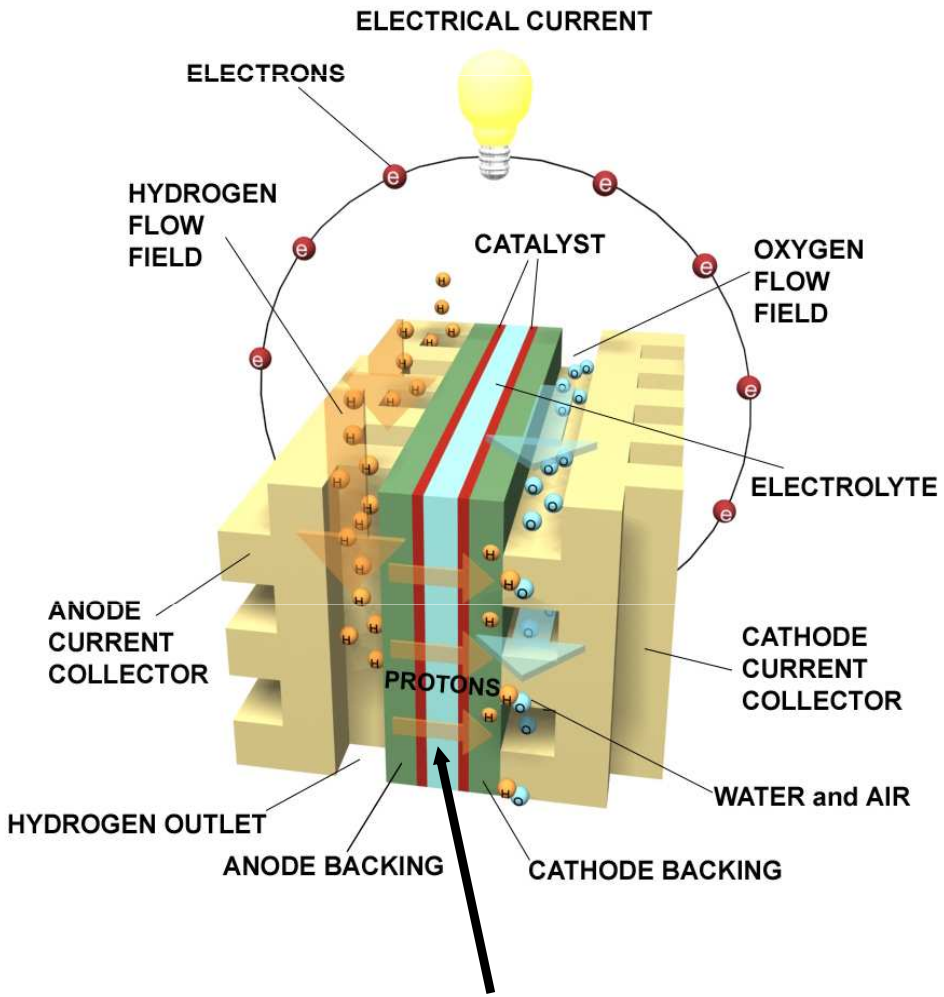
Results – Microbiology

- Antimicrobial effect of zeolites & composites
- Over 50 bacteria , fungi and microbes are being tested



Kamışoğlu, K., Aksoy, E.A.; Akata, B., Hasırcı N., Baç, N. *J. of Appl. Polym. Sci.*, 110, 2854-2861, 2008.

Application of Zeolites in PEM Fuel Cells



membrane

Reactions:

PEMFC

Anode: $H^2 \rightarrow 2H^+ + 2e^-$

Cathode: $\frac{1}{2}O_2 + 2e^- + 2H^+ \rightarrow H_2O$

$\frac{1}{2}O_2 + H_2 \rightarrow H_2O$

DMFC

Anode: $CH_3OH + H_2O \rightarrow CO_2 + 6H^+ + 6e^-$

Cathode: $3/2\ O_2 + 6H^+ + 6e^- \rightarrow 3\ H_2O$

$CH_3OH + 3/2\ O_2 \rightarrow CO_2 + 2\ H_2O$

Polymer Electrolyte Fuel Cells (PEMFC) Applications- Anode, Polymer Electrolyte, Cathode

Limitations with current perfluorosulfonic acid membranes (Nafion):

-Loss of conductivity when dehydrated

-Low operating temp. (80 C)

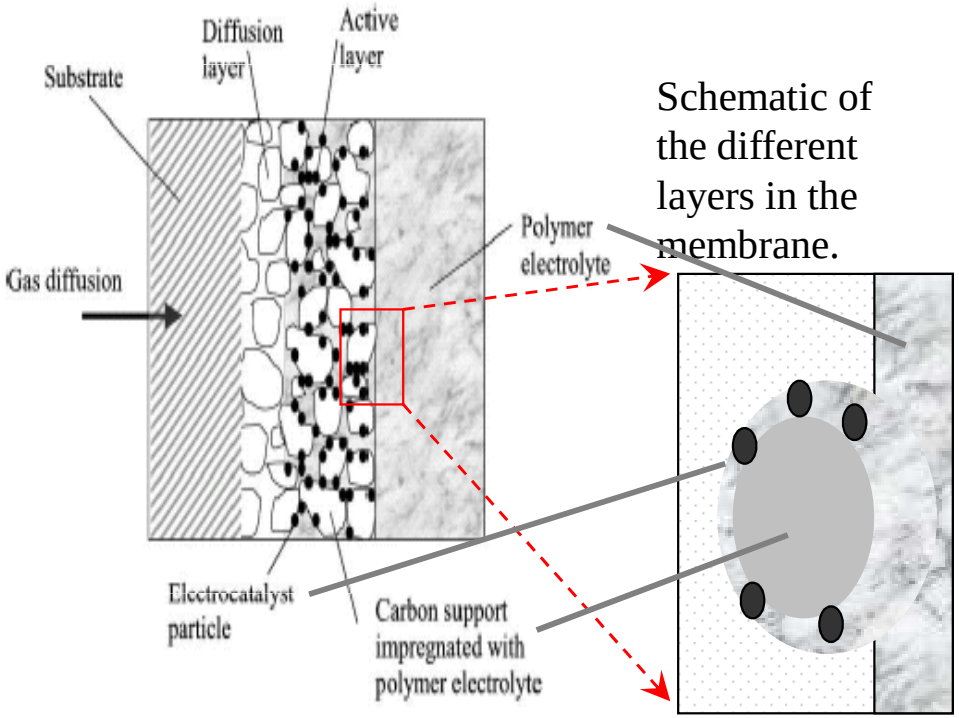
-High cost

Motivation for Elevated Temperature (100°C-200°C) PEM Fuel Cell Operation:

- •Enhanced kinetic rates
- •Lower CO poisoning
- •Improved water and thermal management
- •Alleviate system integration issues

Requirement of New Polymer Electrolyte Membrane for High Performance PEMFC

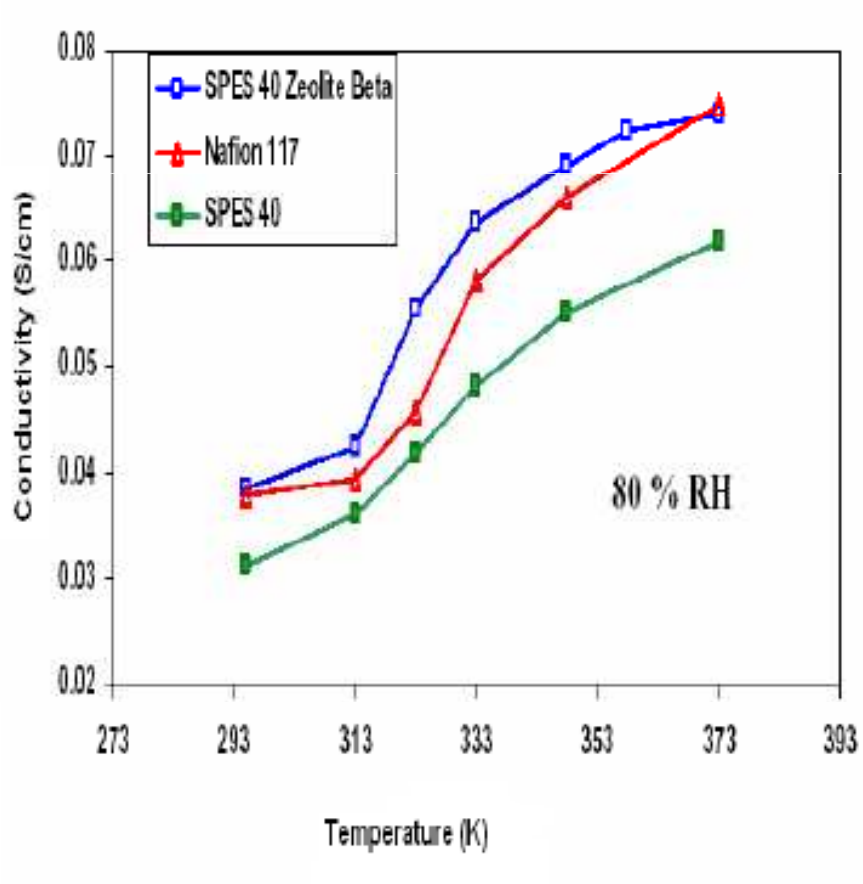
- •Cheap, high Tg temperature and long durability
- •High proton conductivity at elevated temperature and lower relative humidity



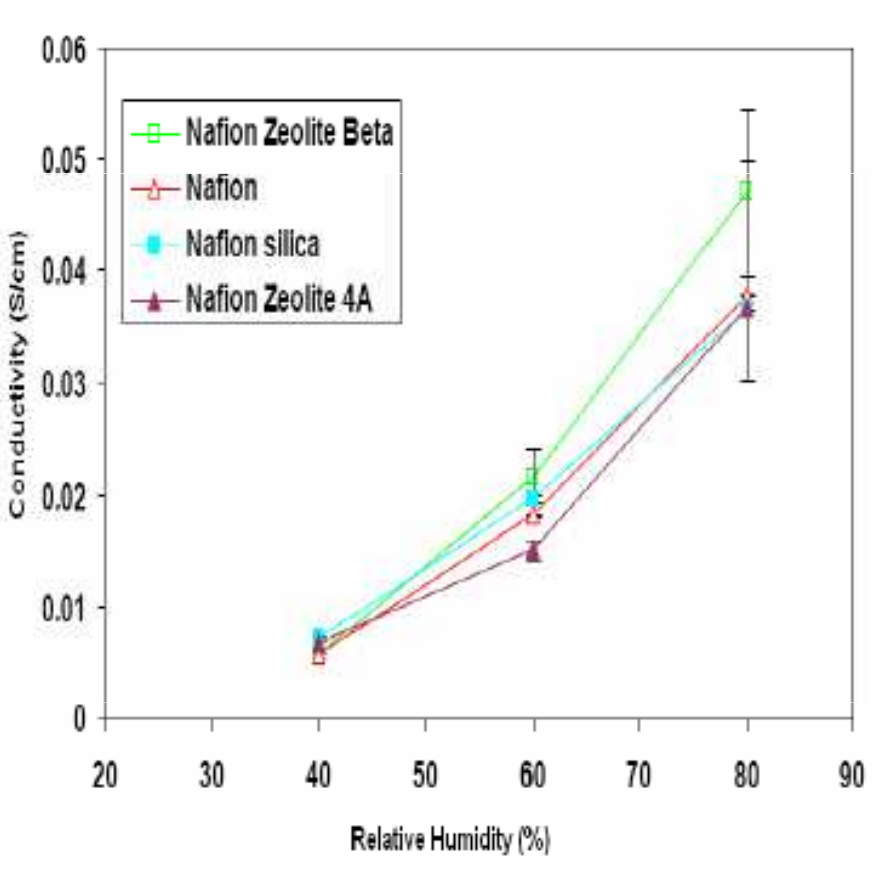
Schematic of the different layers in the membrane.

P. Costamagna, S. Srinivasan, *J. Power Sources*, 102 (2001) 242

Effect of inorganic additives on proton conductivity



(a)



(b)

Bac N. et al. International Hydrogen Energy Conference Istanbul, 13- 15 July 2005

SUMMARY

Knowledge base from space grown zeolites results in spinoffs with new products for the society. Some of these are:

- Antimicrobial Zeolites -
- Nanocomposite zeolite-polymer fuel cell membranes. Portable power, or utilization of hydrogen energy in the future. Helps reduce global warming.
- Microencapsulation of perfume molecules in zeolite nanopores for extended release in detergents/fabric softeners

In memory of the STS-107 Columbia crew (K.Chawla, R.D. Husband, M.P. Anderson L.Clark, I.Ramon, W.C. McCool, D.Brown)

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