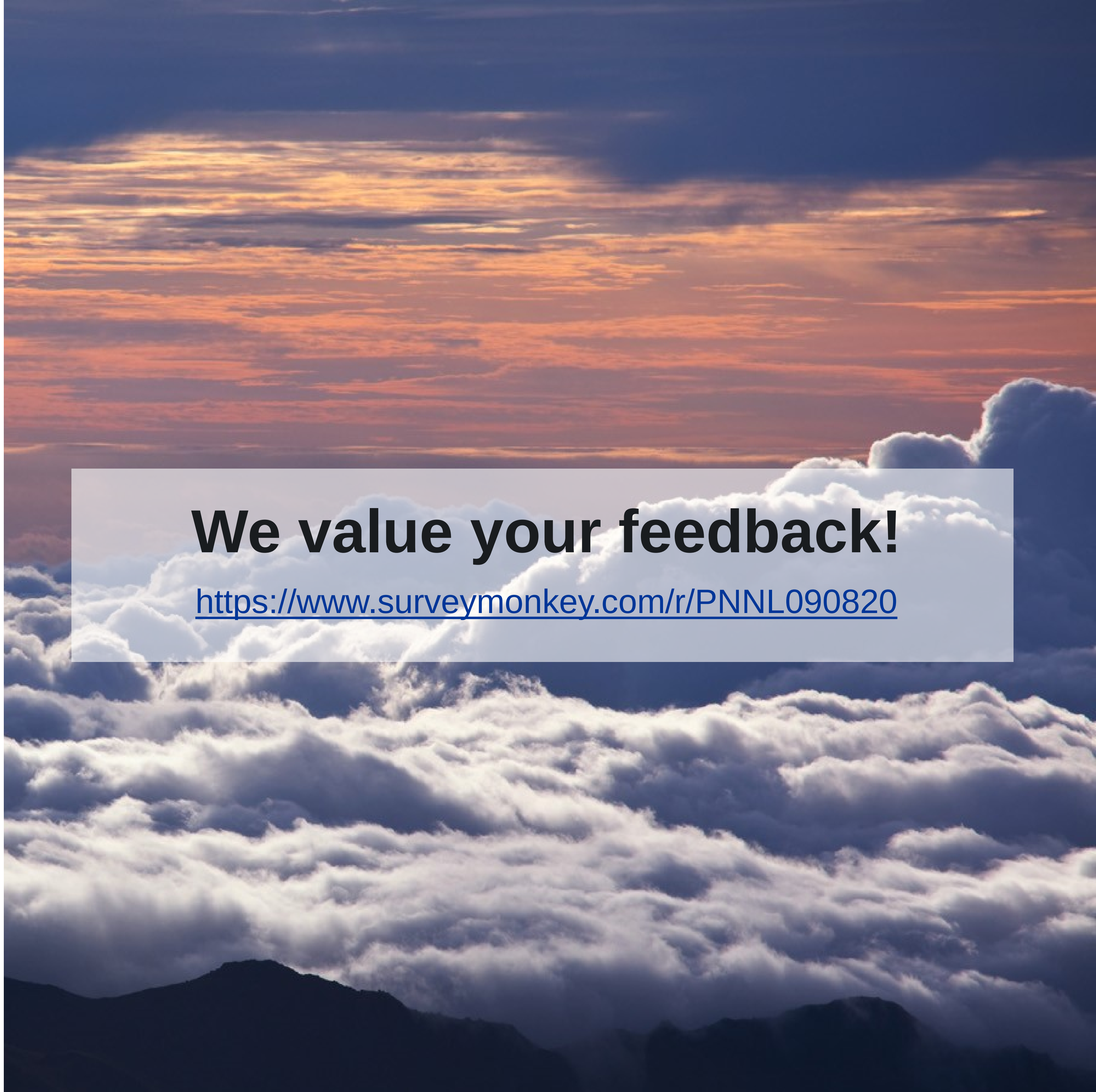
The New Stone Age

Tiffany Kaspar, Materials Scientist
Pacific Northwest National Laboratory



We value your feedback!

https://www.surveymonkey.com/r/PNNL090820

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1 of 17
U.S. DOE Labs

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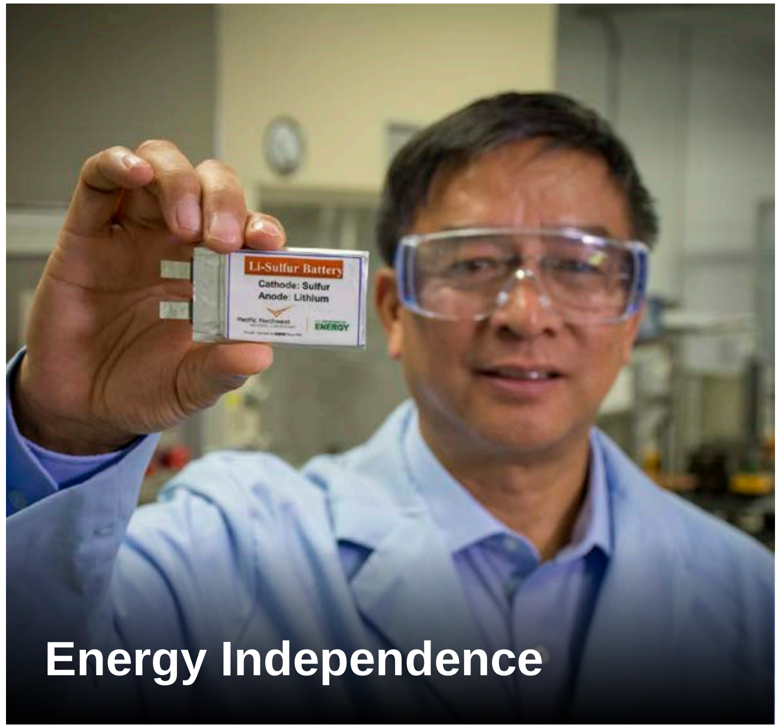
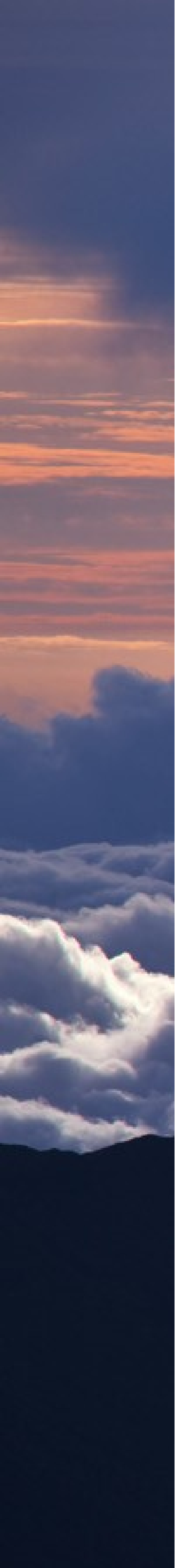
PNNL is Focused on **DOE's MISSIONS** and Addressing Critical **NATIONAL NEEDS**





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PNNL is an ECONOMIC ENGINE

4,722
Employees

265
Inventions

$1.46B
Total Economic Output

$1.01B
Annual Spending

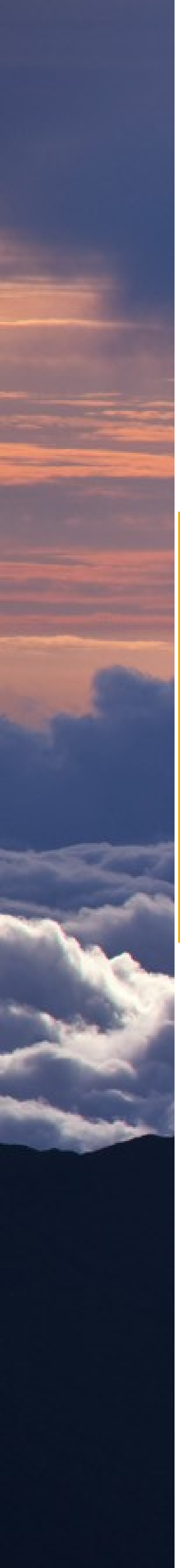
88
Patents

7,180
Jobs Generated
in Washington

$465M
Total Payroll

34
Licenses

193
Companies
with PNNL Roots



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50+ Years

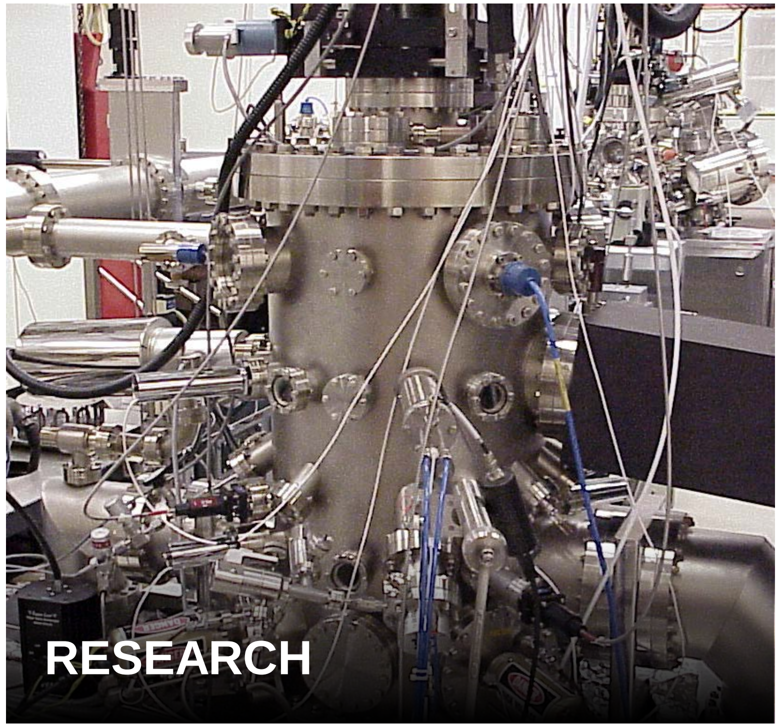
Developing Goodwill

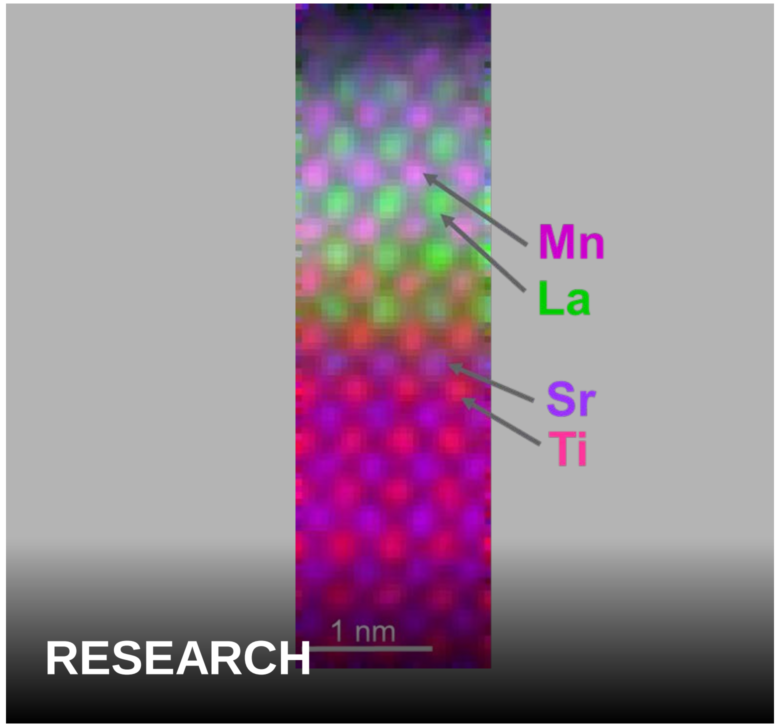
	Philanthropic Investments	Team Battelle Volunteer Hours	Community Organizations
Decades	$28.5M	347,000	>120
FY19	$0.52M	30,000	56

A little about me…

Tiffany Kaspar

- B.S. Chemical Engineering, CU-Boulder, 1998
- Ph.D. Chemical Engineering, UW, 2004
- At PNNL since 2000





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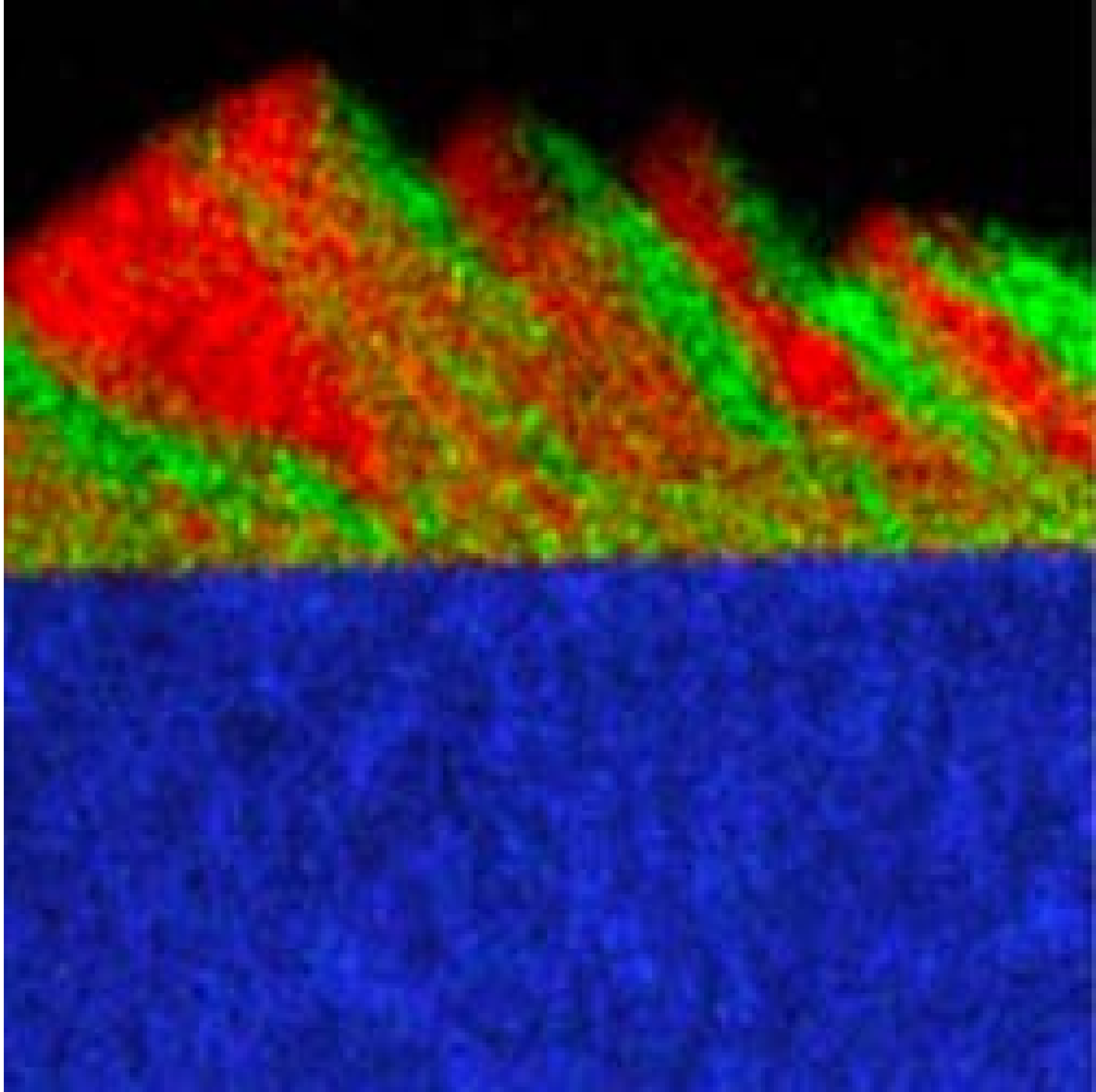
The New Stone Age

September 9, 2020

Tiffany Kaspar
Senior Research Scientist
Atomically Precise Materials Group
Materials Sciences Division

U.S. DEPARTMENT OF ENERGY   BATTELLE

PNNL is operated by Battelle for the U.S. Department of Energy



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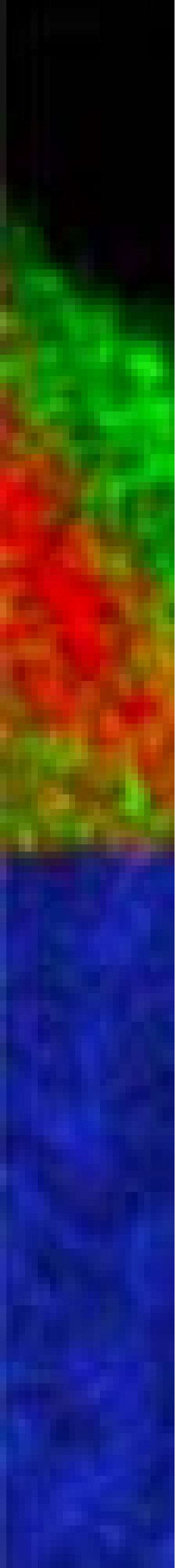
Hypothesis:

Oxides (in the form of rocks) began human technological development

And

Oxides (in the form of advanced materials precisely synthesized at the atomic scale) will soon…

SAVE THE WORLD!!



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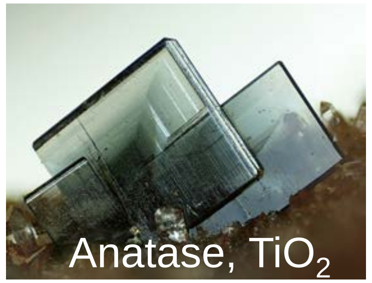
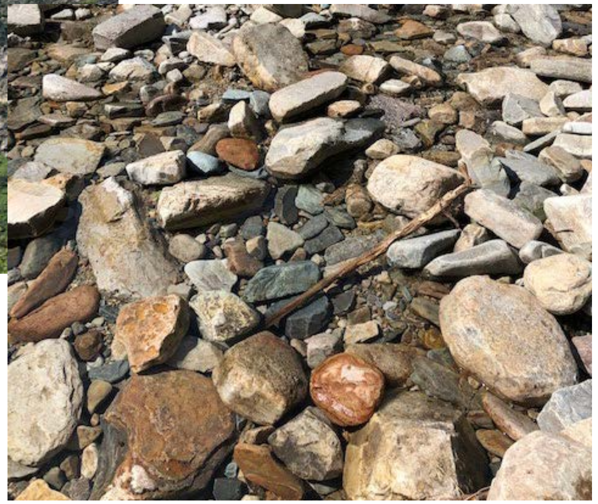
What are oxides?

https://www.mindat.org/



Minerals: metal + oxygen; defined composition and crystal structure

Cabinet Mountains, MT



Rocks

Ceramics

Glass

https://theglasspunty.com/

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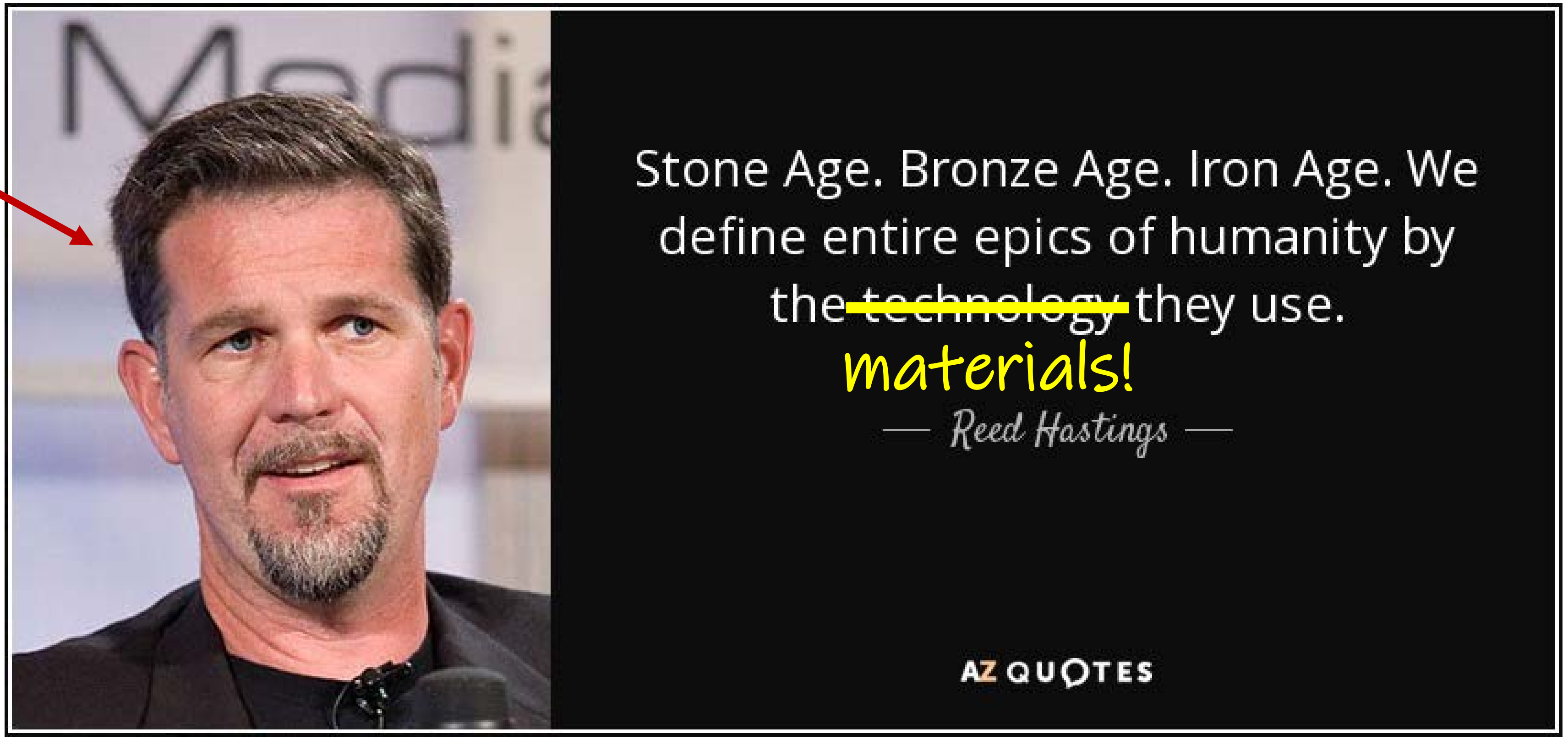
NETFLIX
co-founder



https://www.azquotes.com/quote/918949

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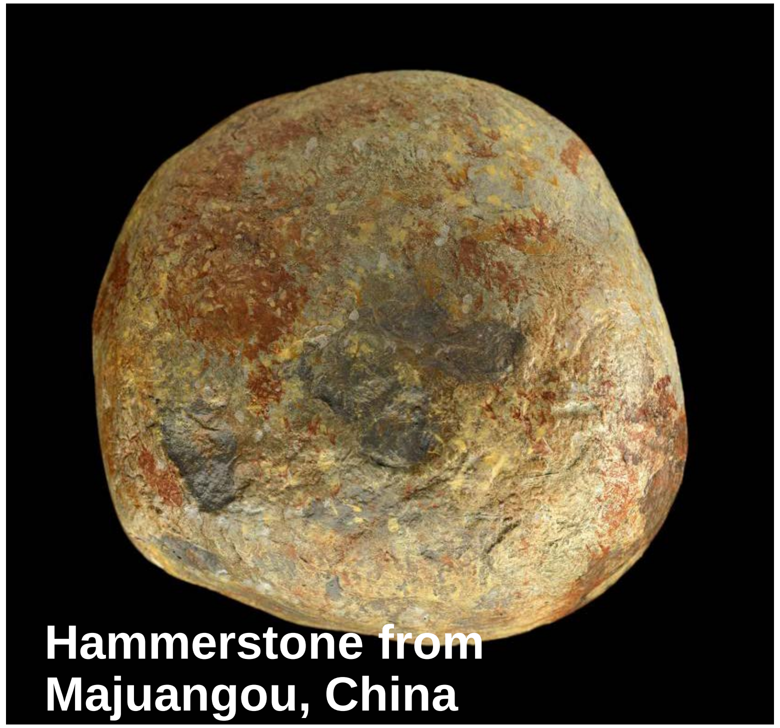
The (Original) Stone Age began 2.6 million years ago

- Which stone to use?
- Engineering advances required new materials
  - Bone, ivory, antler…

Early on…


Hammerstone from Majuangou, China


Handaxe from Bose, China

Later…


Stone graver from La Madeleine, Dordogne, France


Katanda bone harpoon point, Central Africa

Smithsonian: https://humanorigins.si.edu/evidence/behavior/stone-tools

Stone through history: structural

Ancient Roman aqueduct, Segovia, Spain

Chartres Cathedral, Chartres, France

Independence Hall, Philadelphia, PA

Mesa Verde National Park, CO

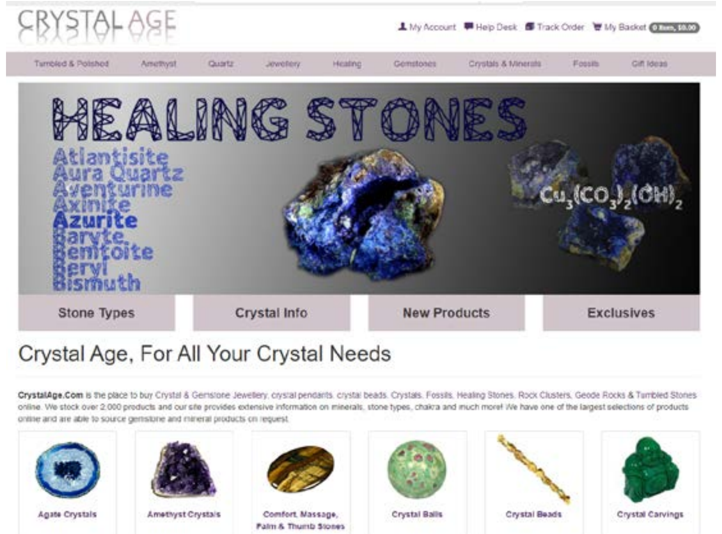
The Salk Institute, La Jolla, CA

Stone through history: functional

Magnetism

Lodestone (magnetite) compass, China, 220 BCE
https://www.smith.edu/hsc/museum/ancient_inventions/compass2.html

“Medicine”



Modern website selling crystals for “healing”
https://www.crystalage.com/

Electrical insulation

Large ceramic insulators on high voltage power lines
https://www.quora.com/What-are-the-cones-on-power-lines

Far fewer functional uses than structural…

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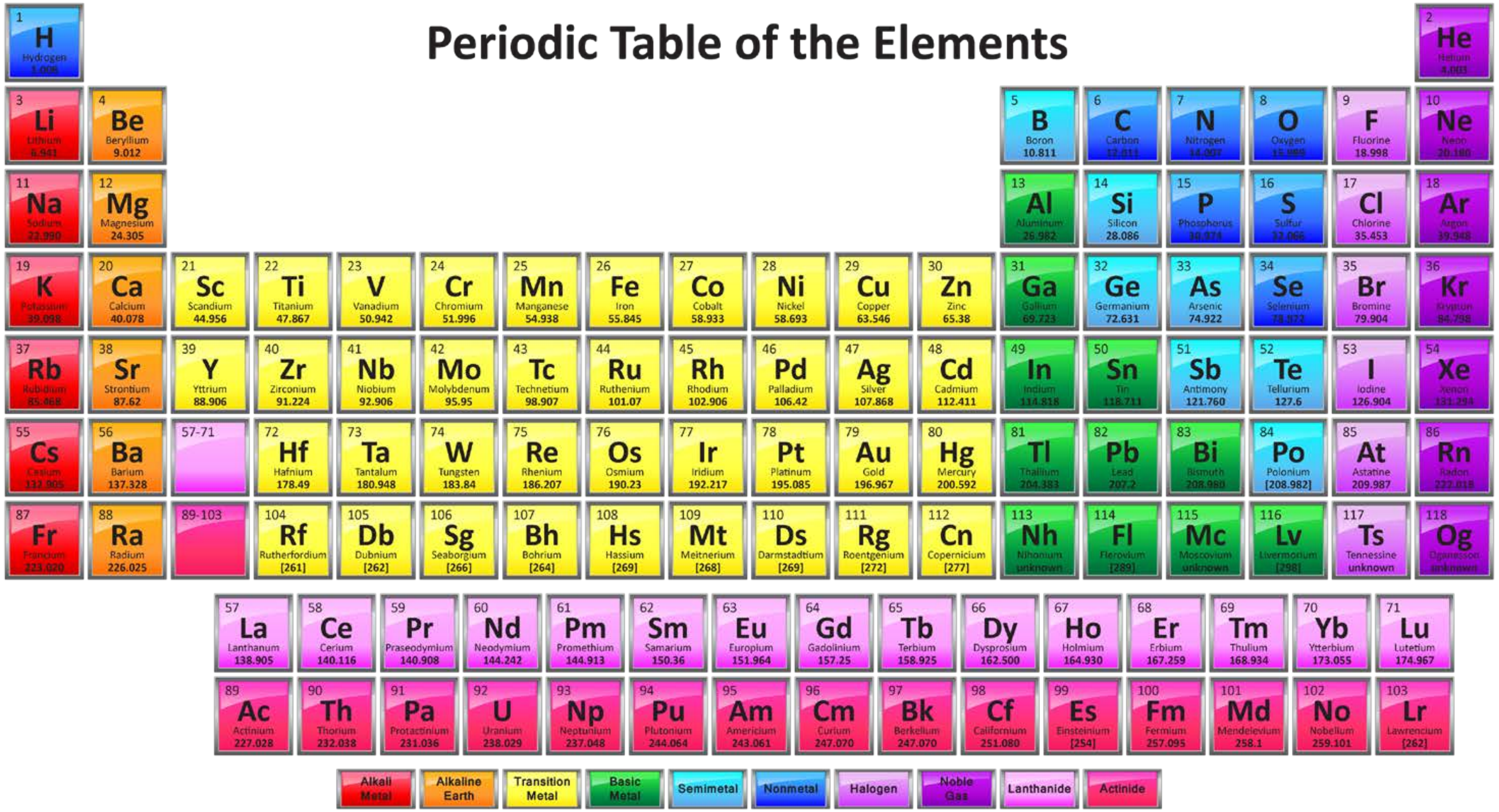
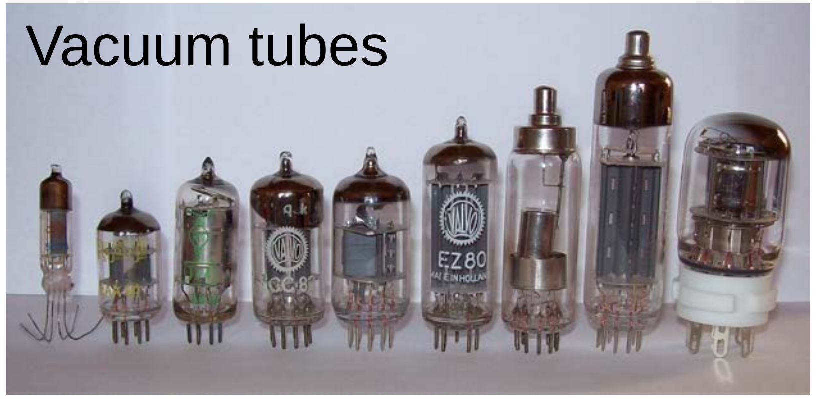
Post-WW II: Materials *engineering* became materials *science*



- Scientists learned to purify materials and put them together in new ways
- The periodic table of elements became a playground to invent new materials
- Materials science led to huge advances in both science and technology

The oxide Renaissance



Vacuum tube diodes were invented in the early 1900’s as a component of the first electronic circuits
- Bulky, delicate

Diode: allows electrical current to flow in one direction only

1926

J.E. Lillienfeld patents a solid-state field-effect transistor idea

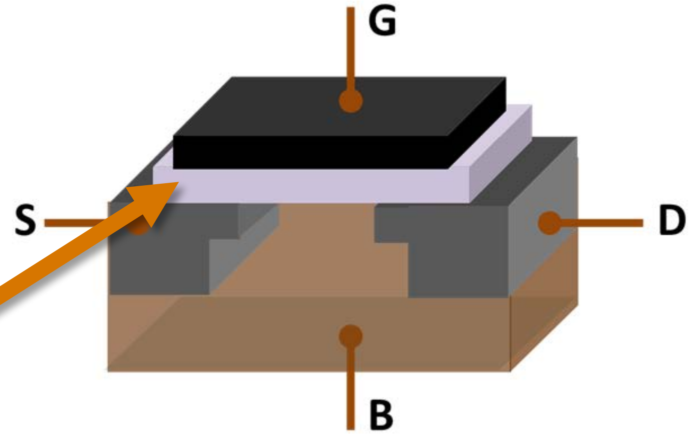
High purity semiconductors

1947

Bell Labs invents first solid-state point-contact transistor

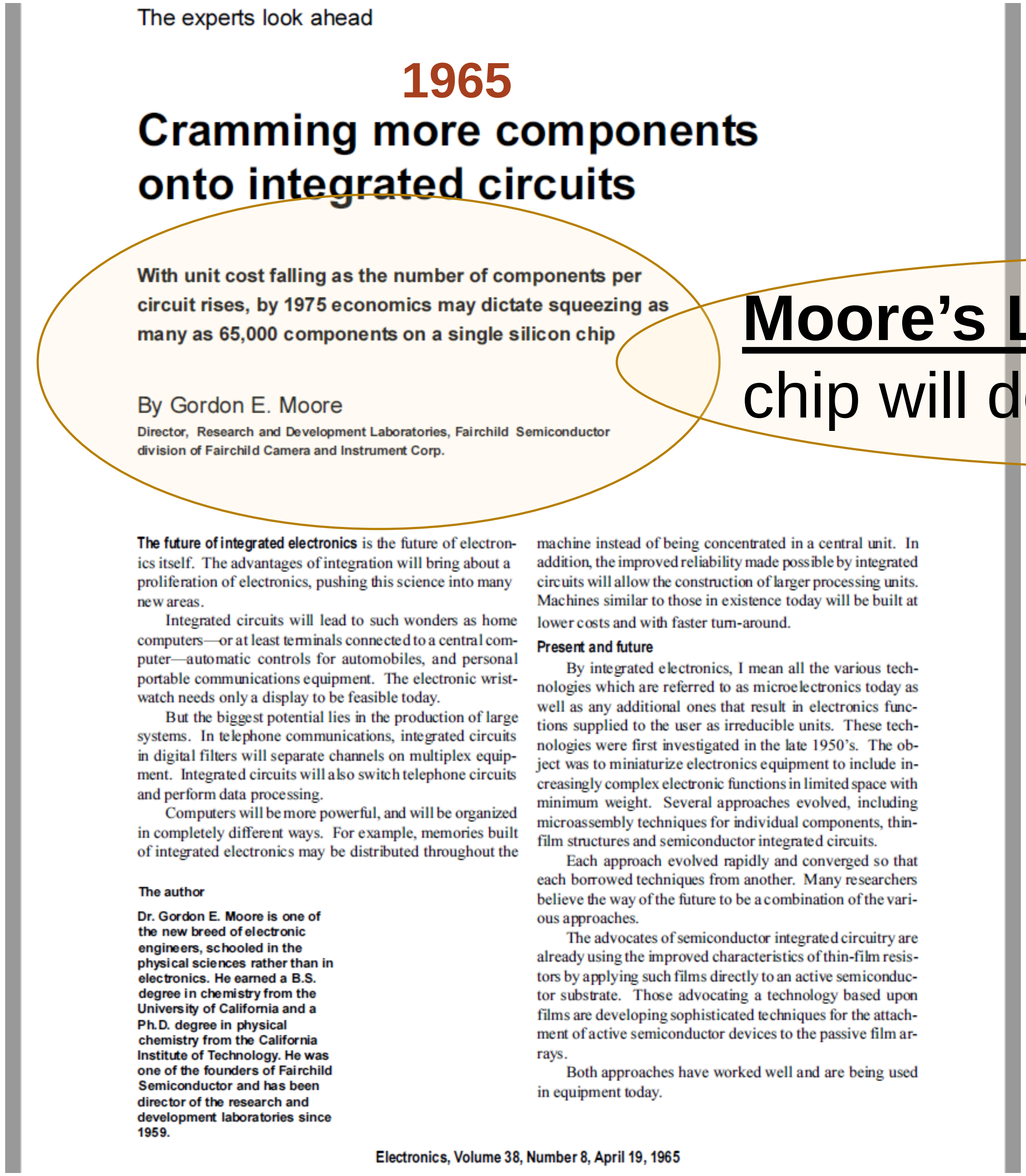
SiO_2 oxide layer on silicon semiconductor

1959



Bell Labs invents first solid-state field-effect transistor

The oxide Renaissance



The experts look ahead

1965

Cramming more components onto integrated circuits

With unit cost falling as the number of components per circuit rises, by 1975 economics may dictate squeezing as many as 65,000 components on a single silicon chip

By Gordon E. Moore

Director, Research and Development Laboratories, Fairchild Semiconductor division of Fairchild Camera and Instrument Corp.

The future of integrated electronics is the future of electronics itself. The advantages of integration will bring about a proliferation of electronics, pushing this science into many new areas.

Integrated circuits will lead to such wonders as home computers—or at least terminals connected to a central computer—automatic controls for automobiles, and personal portable communications equipment. The electronic wristwatch needs only a display to be feasible today.

But the biggest potential lies in the production of large systems. In telephone communications, integrated circuits in digital filters will separate channels on multiplex equipment. Integrated circuits will also switch telephone circuits and perform data processing.

Computers will be more powerful, and will be organized in completely different ways. For example, memories built of integrated electronics may be distributed throughout the machine instead of being concentrated in a central unit. In addition, the improved reliability made possible by integrated circuits will allow the construction of larger processing units. Machines similar to those in existence today will be built at lower costs and with faster turn-around.

Present and future

By integrated electronics, I mean all the various technologies which are referred to as microelectronics today as well as any additional ones that result in electronics functions supplied to the user as irreducible units. These technologies were first investigated in the late 1950's. The object was to miniaturize electronics equipment to include increasingly complex electronic functions in limited space with minimum weight. Several approaches evolved, including microassembly techniques for individual components, thin-film structures and semiconductor integrated circuits.

Each approach evolved rapidly and converged so that each borrowed techniques from another. Many researchers believe the way of the future to be a combination of the various approaches.

The advocates of semiconductor integrated circuitry are already using the improved characteristics of thin-film resistors by applying such films directly to an active semiconductor substrate. Those advocating a technology based upon films are developing sophisticated techniques for the attachment of active semiconductor devices to the passive film arrays.

Both approaches have worked well and are being used in equipment today.

The author

Dr. Gordon E. Moore is one of the new breed of electronic engineers, schooled in the physical sciences rather than in electronics. He earned a B.S. degree in chemistry from the University of California and a Ph.D. degree in physical chemistry from the California Institute of Technology. He was one of the founders of Fairchild Semiconductor and has been director of the research and development laboratories since 1959.

Electronics, Volume 38, Number 8, April 19, 1965

<u>Moore's Law</u>: transistors per chip will double every two years

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Electronics, Volume 38, Number 8, April 19, 1965

Gordon Moore:

Ph.D. degree in physical chemistry

Robert Noyce:

Ph.D. degree in physics

1968:

Moore and Noyce co-founded Intel Corp.

The oxide Renaissance

The experts look ahead

1965

Cramming more components

Chemistry + physics = materials science!

Computer technology advances by understanding and controlling the *materials* of the chip(s)

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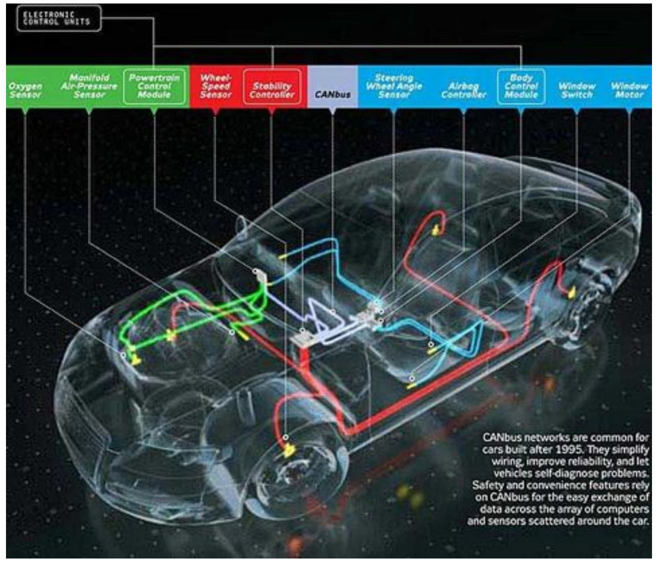
Gordon Moore:

Ph.D. degree in physical chemistry

The oxide Renaissance

Everything Gordon Moore foresaw for “integrated electronics” has come to pass!

https://www.popularmechanics.com/cars/how-to/a7386/how-it-works-the-computer-inside-your-car/



https://www.tesla.com/autopilot

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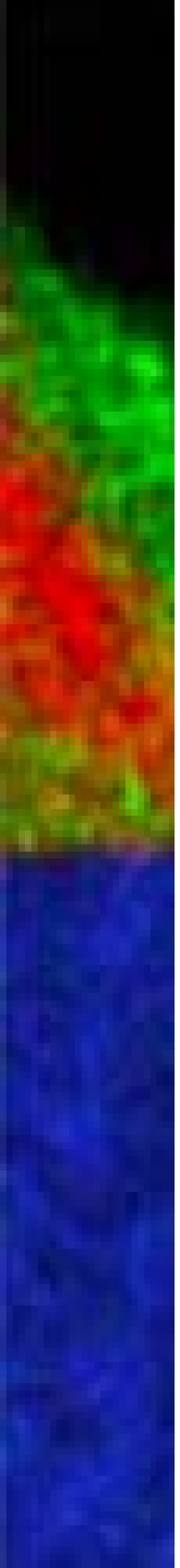
The oxide Renaissance

Smartphones use **at least 70** of the 83 non-radioactive elements!



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ELEMENTS OF A SMARTPHONE

ELEMENTS COLOUR KEY: ALKALI METAL · ALKALINE EARTH METAL · TRANSITION METAL · GROUP 13 · GROUP 14 · GROUP 15 · GROUP 16 · HALOGEN · LANTHANIDE

SCREEN

49 In Indium · 8 O Oxygen · 50 Sn Tin

Indium tin oxide is a mixture of indium oxide and tin oxide, used in a transparent film in the screen that conducts electricity. This allows the screen to function as a touch screen.

13 Al Aluminium · 14 Si Silicon · 8 O Oxygen · 19 K Potassium

The glass used on the majority of smartphones is an aluminosilicate glass, composed of a mix of alumina (Al_2O_3) and silica (SiO_2). This glass also contains potassium ions, which help to strengthen it.

39 Y Yttrium · 57 La Lanthanum · 65 Tb Terbium · 59 Pr Praseodymium · 63 Eu Europium · 66 Dy Dysprosium · 64 Gd Gadolinium

A variety of Rare Earth Element compounds are used in small quantities to produce the colours in the smartphone's screen. Some compounds are also used to reduce UV light penetration into the phone.

ELECTRONICS

29 Cu Copper · 47 Ag Silver · 79 Au Gold · 73 Ta Tantalum

Copper is used for wiring in the phone, whilst copper, gold and silver are the major metals from which microelectrical components are fashioned. Tantalum is the major component of micro-capacitors.

28 Ni Nickel · 66 Dy Dysprosium · 59 Pr Praseodymium · 65 Tb Terbium · 60 Nd Neodymium · 64 Gd Gadolinium

Nickel is used in the microphone as well as for other electrical connections. Alloys including the elements praseodymium, gadolinium and neodymium are used in the magnets in the speaker and microphone. Neodymium, terbium and dysprosium are used in the vibration unit.

14 Si Silicon · 8 O Oxygen · 51 Sb Antimony · 33 As Arsenic · 15 P Phosphorus · 31 Ga Gallium

Pure silicon is used to manufacture the chip in the phone. It is oxidised to produce non-conducting regions, then other elements are added in order to allow the chip to conduct electricity.

50 Sn Tin · 82 Pb Lead

Tin & lead are used to solder electronics in the phone. Newer lead-free solders use a mix of tin, copper and silver.

BATTERY

3 Li Lithium · 27 Co Cobalt · 8 O Oxygen · 6 C Carbon · 13 Al Aluminium

The majority of phones use lithium ion batteries, which are composed of lithium cobalt oxide as a positive electrode and graphite (carbon) as the negative electrode. Some batteries use other metals, such as manganese, in place of cobalt. The battery's casing is made of aluminium.

CASING

6 C Carbon · 12 Mg Magnesium · 35 Br Bromine · 28 Ni Nickel

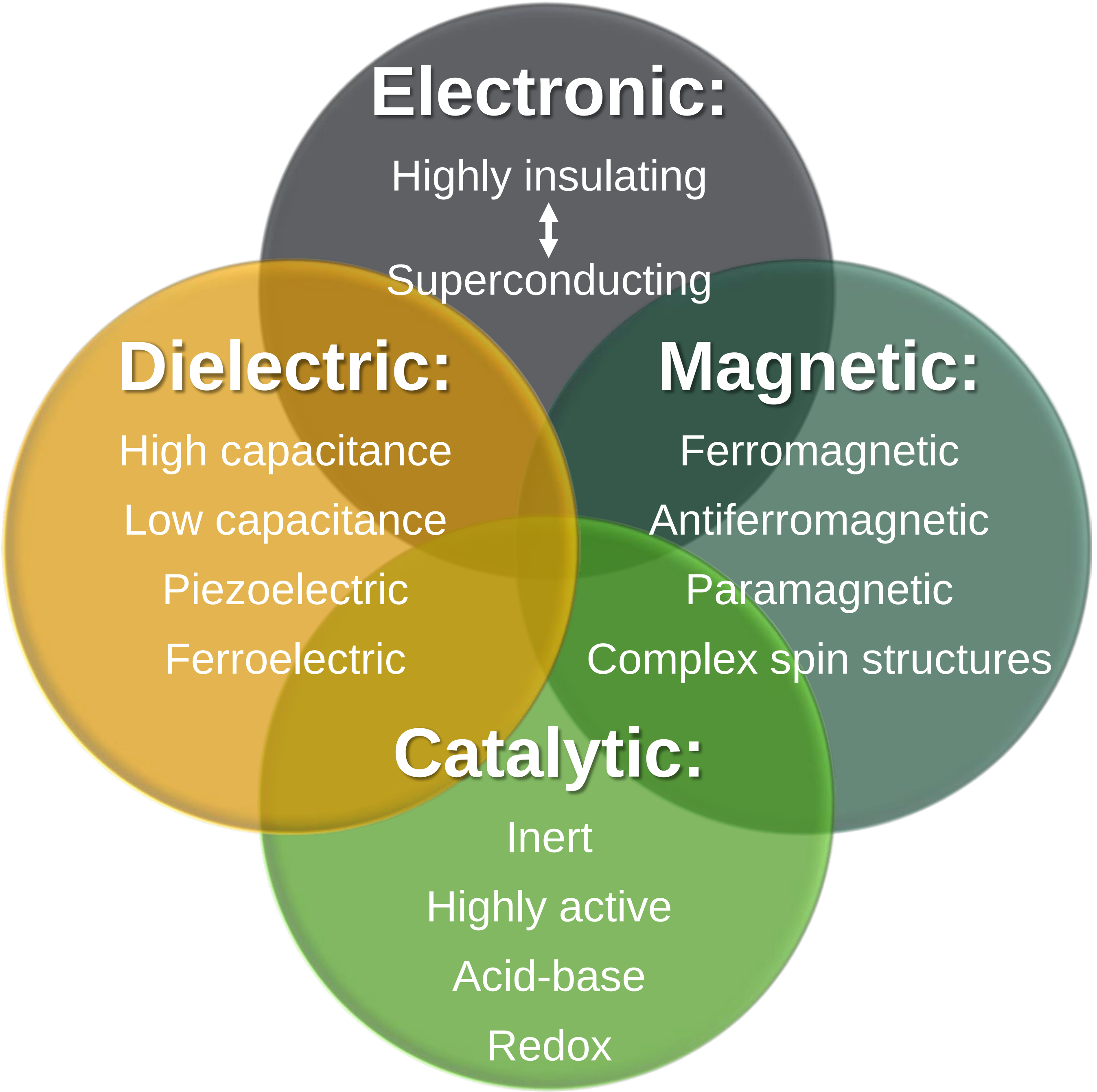
Magnesium compounds are alloyed to make some phone cases, whilst many are made of plastics. Plastics will also include flame retardant compounds, some of which contain bromine, whilst nickel can be included to reduce electromagnetic interference.

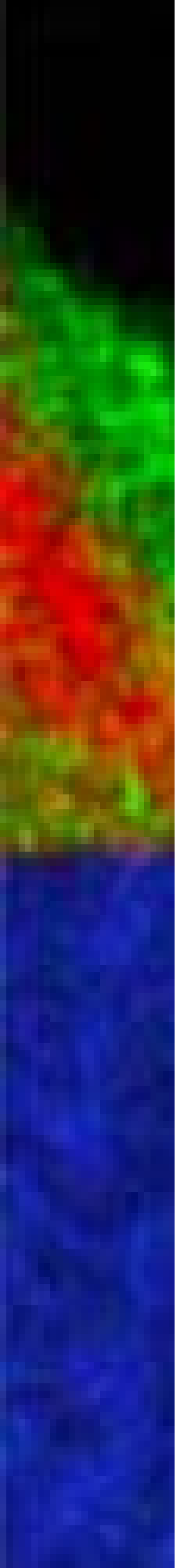
https://www.compoundchem.com/2014/02/19/the-chemical-elements-of-a-smartphone/

Modern functional oxides

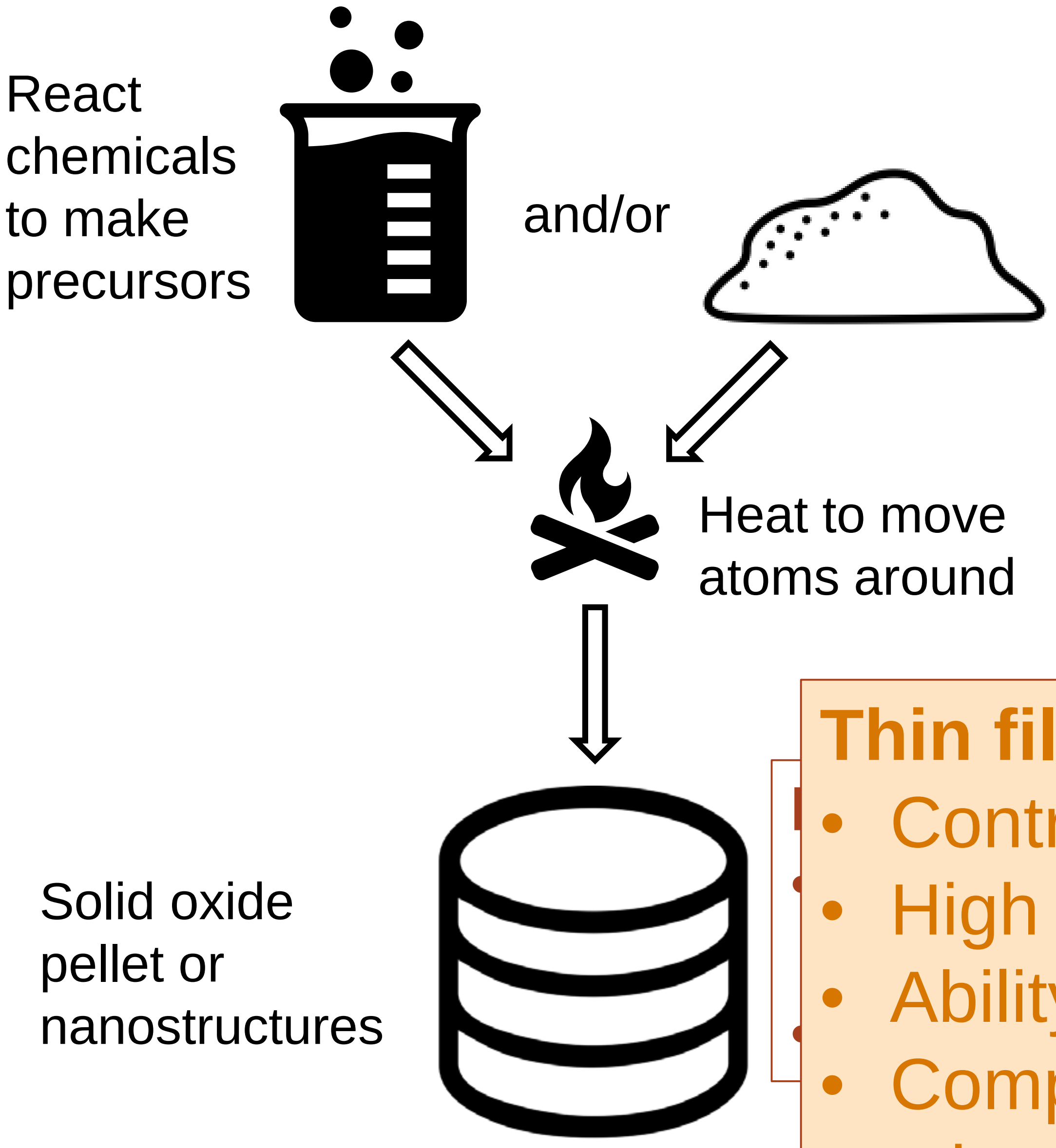
What we have learned in the last ~70 years:

Oxides have the widest range of "functional properties" of any class of materials

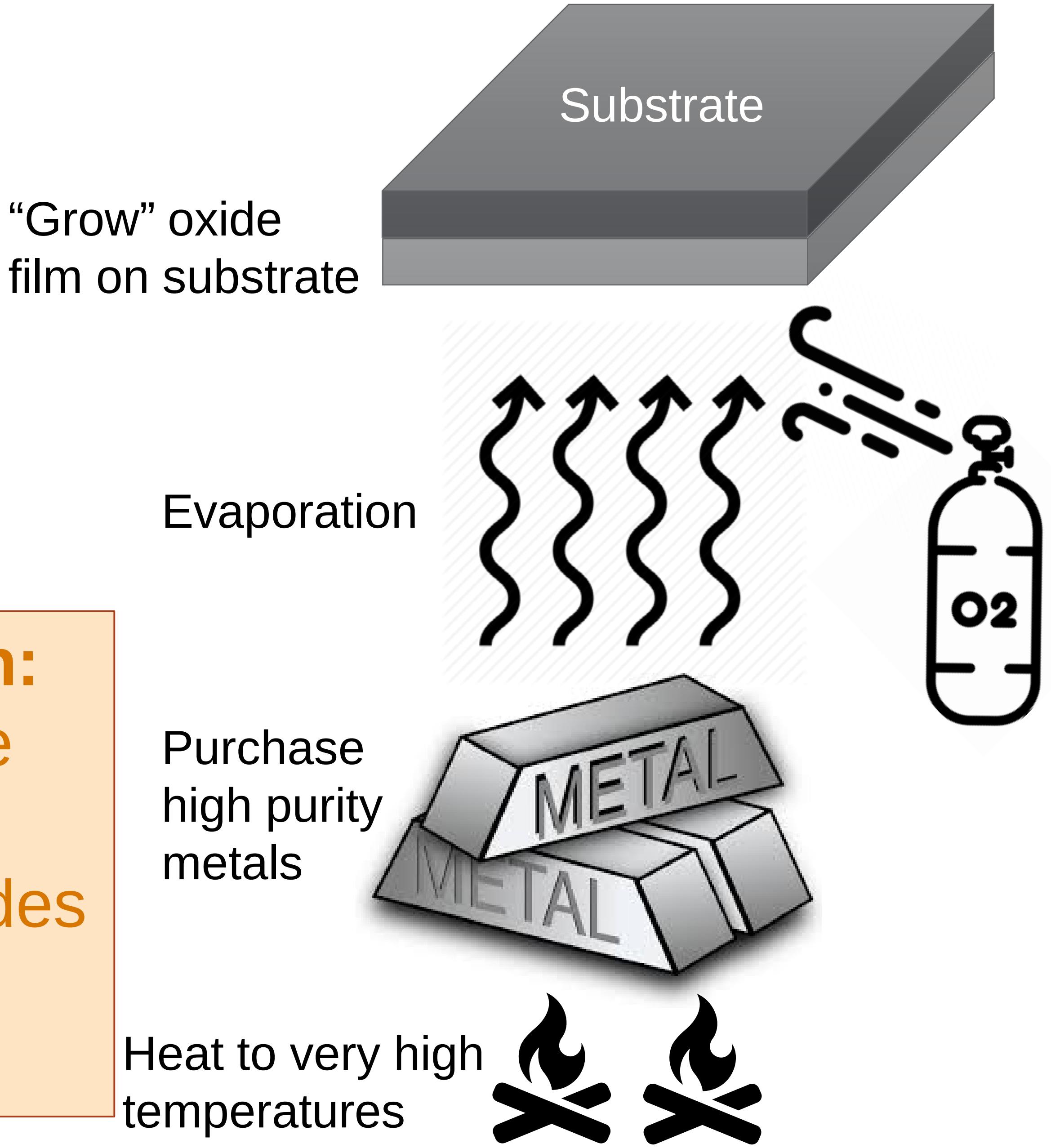




How do we make oxides to study?

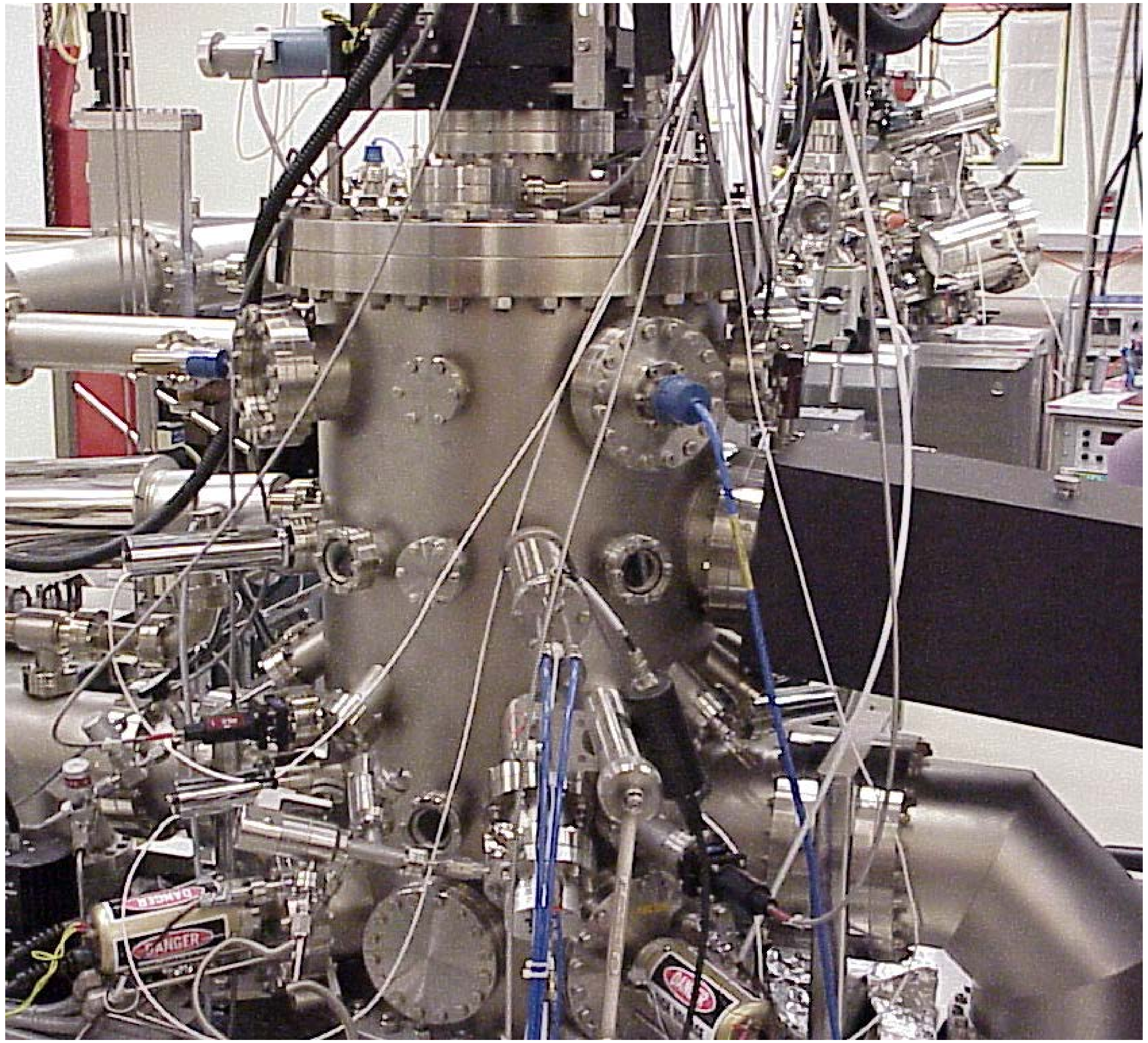


A better way:



Thin film deposition:
- Control over phase
- High purity
- Ability to stack oxides
- Compatible with microelectronics

Molecular beam epitaxy



Ultrahigh vacuum chamber for MBE

High purity oxides

↓

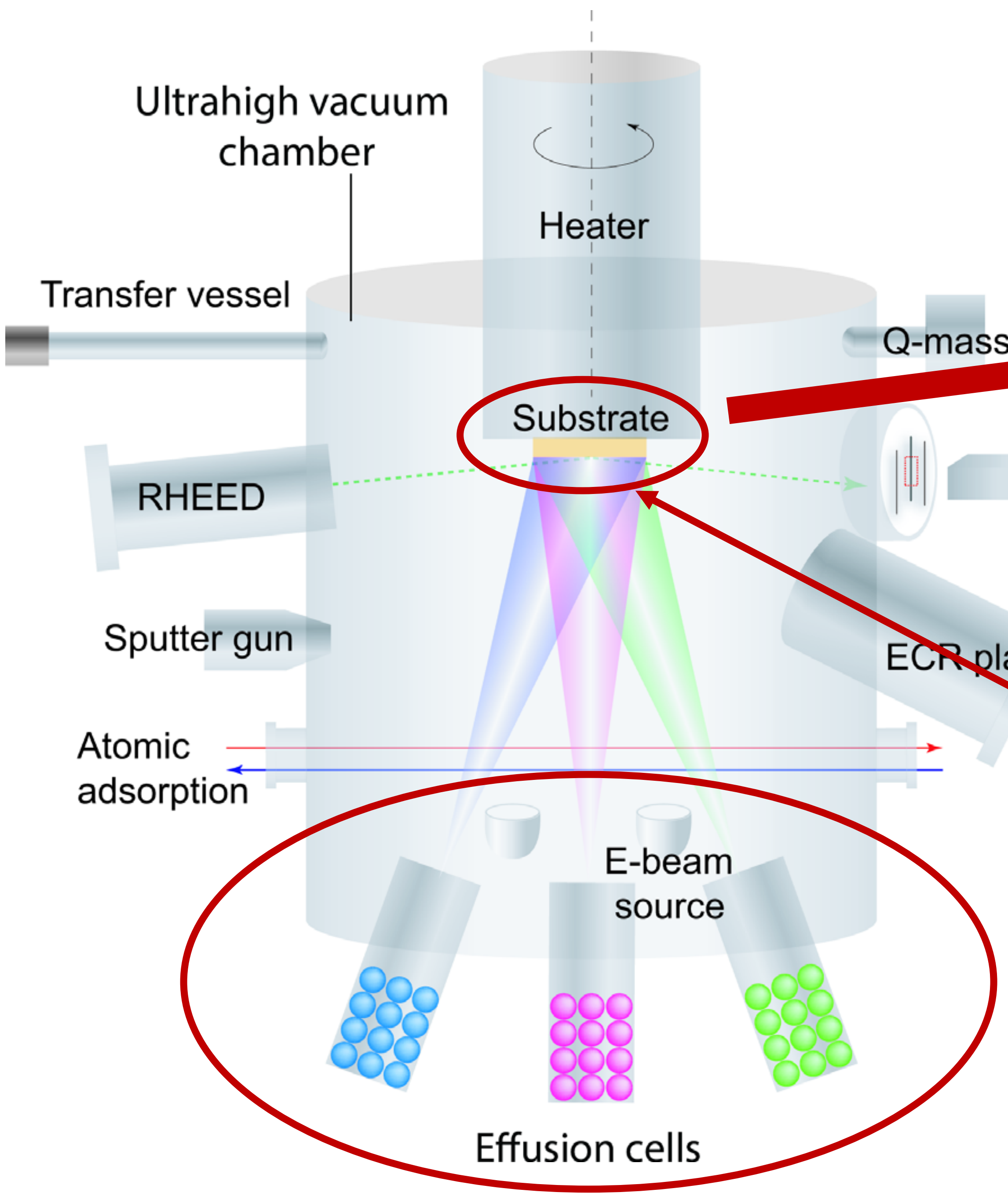
Ultraclean environment

↓

"Ultrahigh vacuum" chamber

~15 pounds (~ 2 gallon jugs of milk) pressing on every square inch!

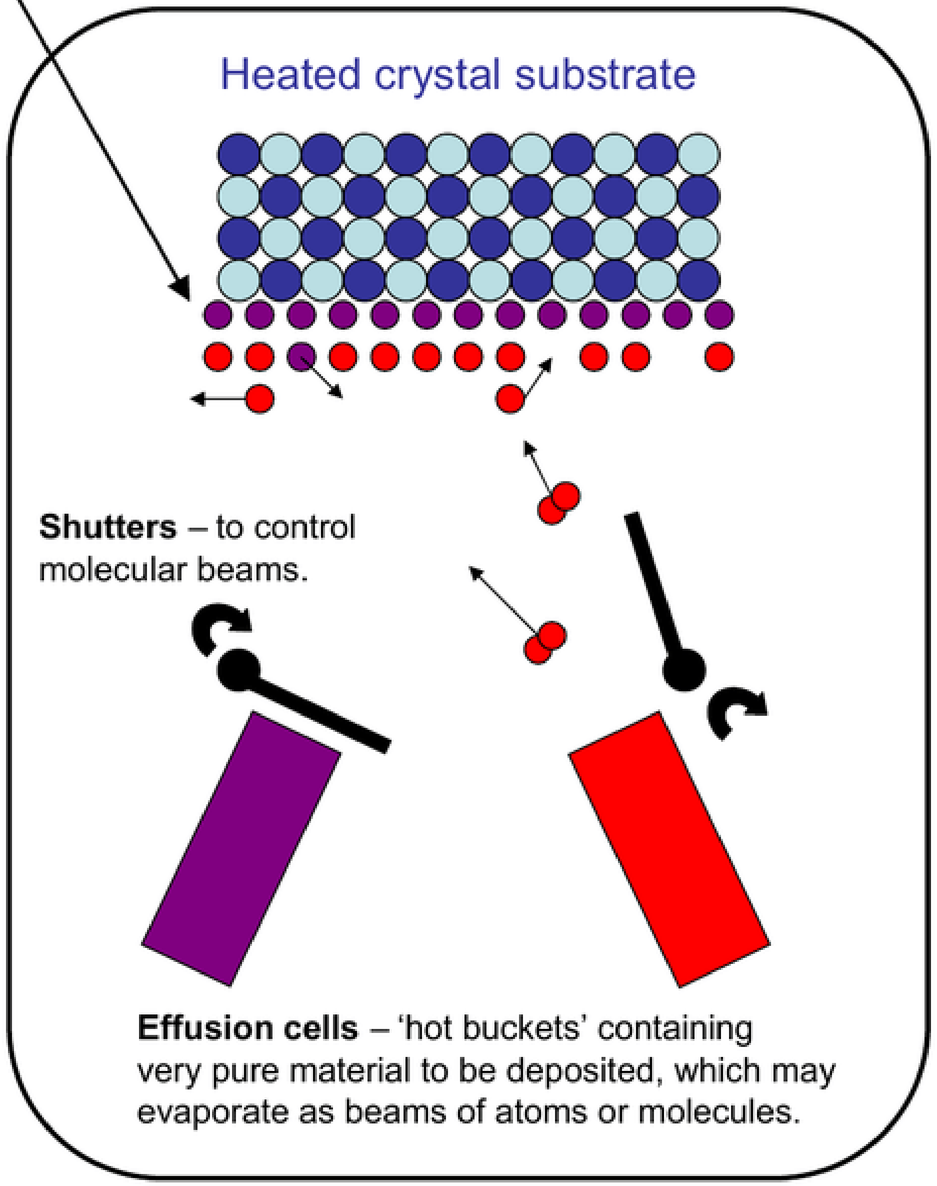
Molecular beam epitaxy



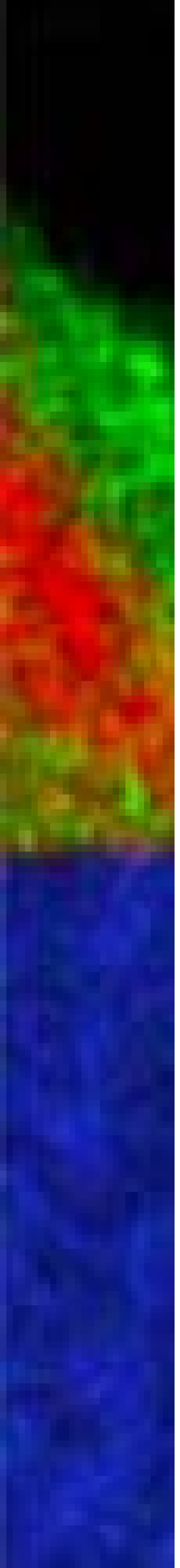
Deposited epitaxial thin film

Oxygen

Metal sources in evaporators



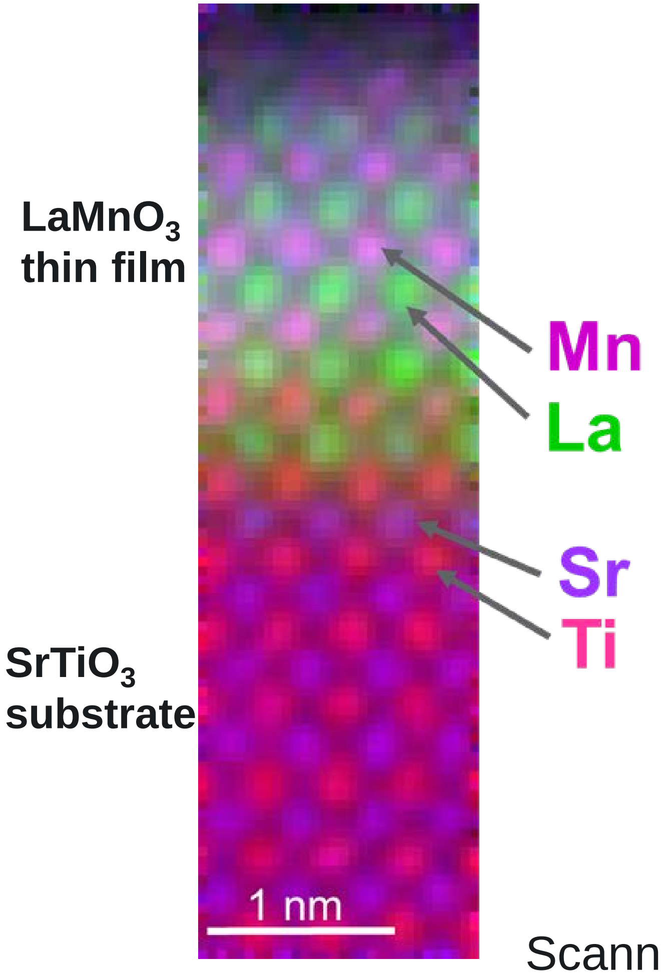
Dr. Gavin Bell, University of Warwick, www.warwick.ac.uk/go/grbell

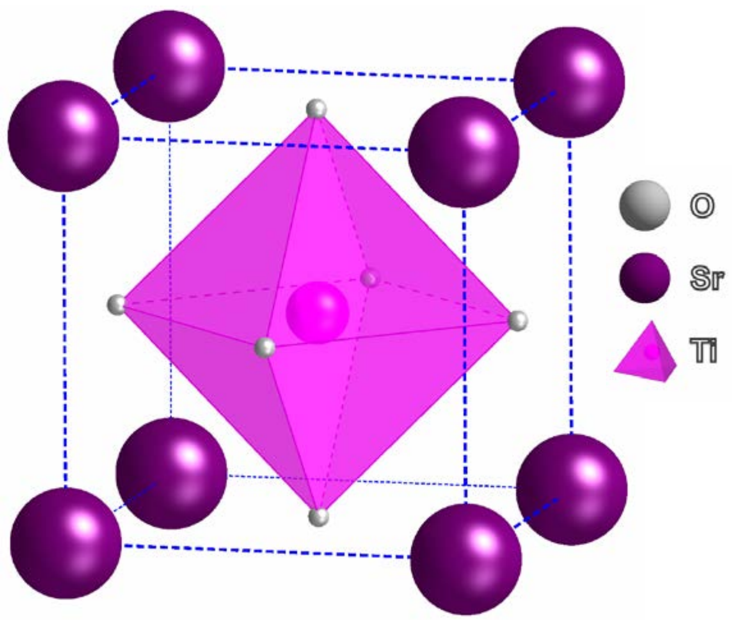


Atomically precise deposition

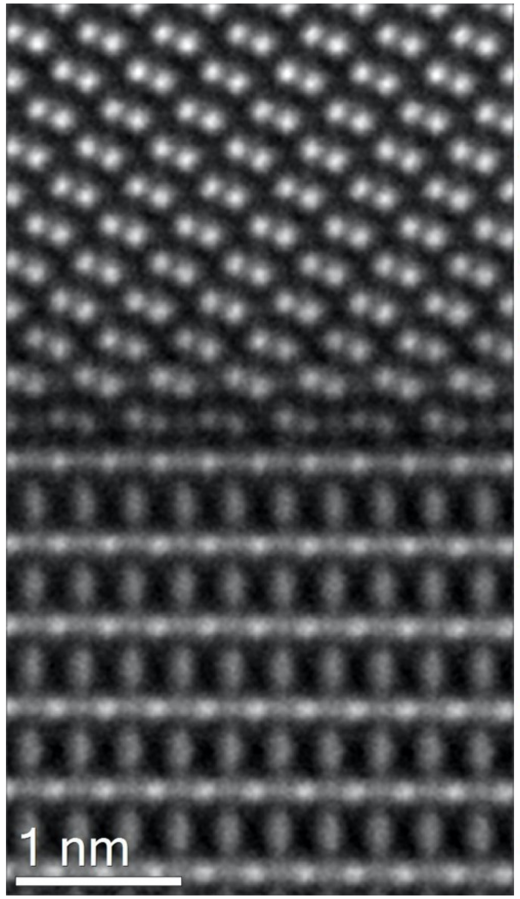
Epitaxy:

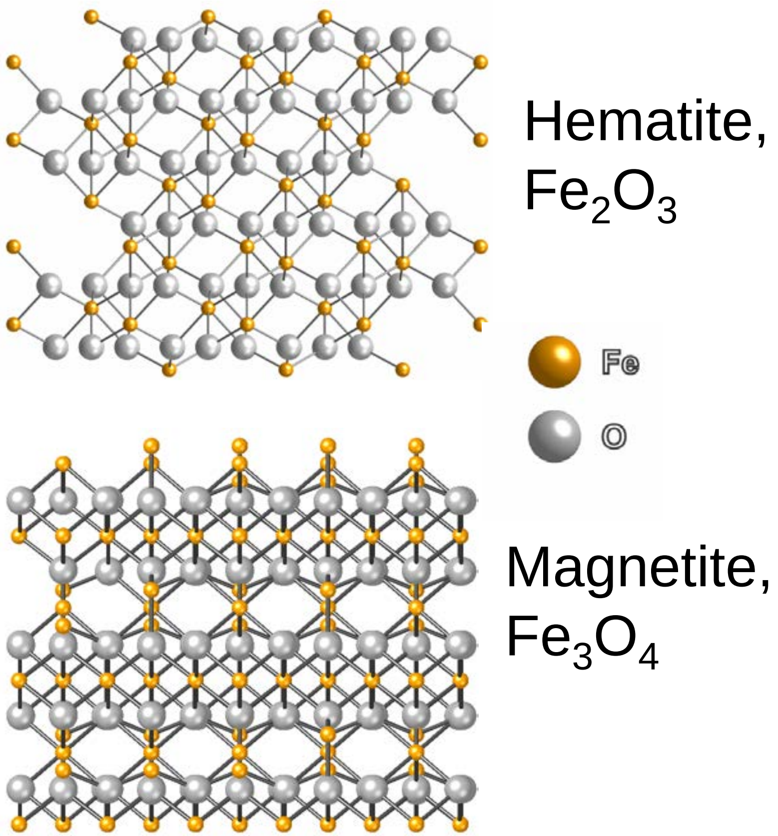
The natural or artificial growth of crystals on a crystalline substrate that controls the structure of the thin film.



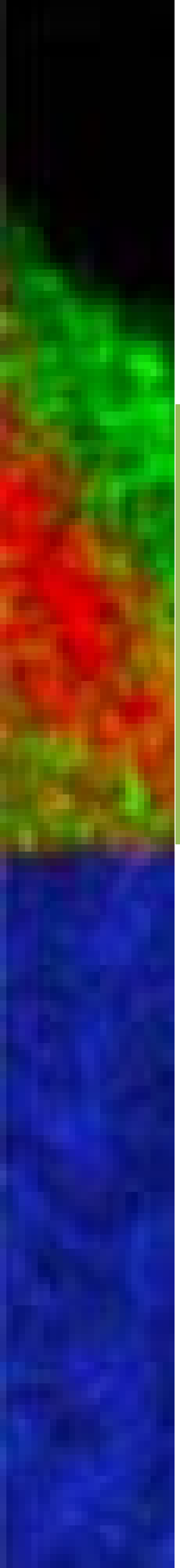


Perovskite crystal structure
$SrTiO_3$, $LaMnO_3$





Scanning transmission electron microscopy (STEM) images



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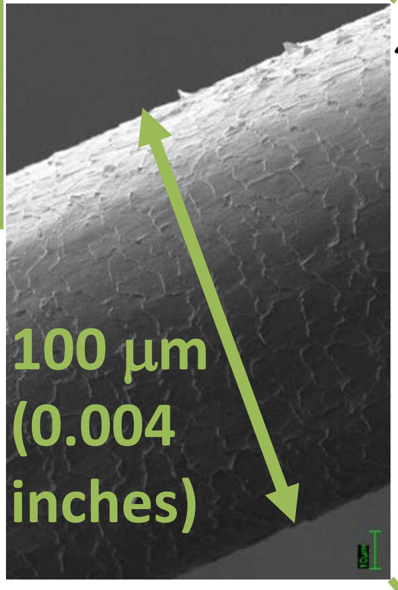
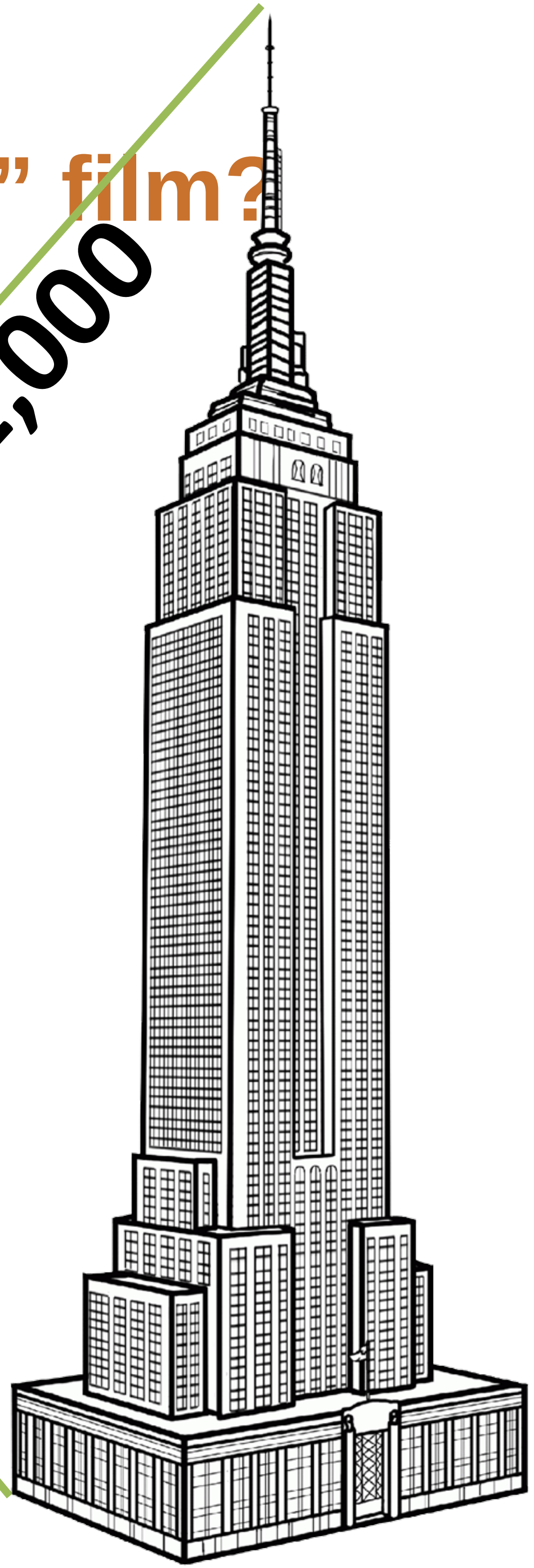
How thin is a "thin" film?

Thin films are ~10,000 times thinner than a human hair

Hair



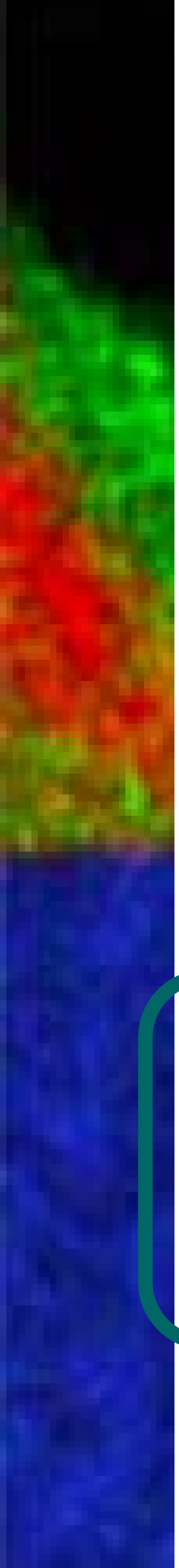
x 4,362,000



Empire State Building: 102 stories, 1,454 ft tall

Thin film:
10 nm thick
(0.01 µm)

1 ¾ inches



Modern functional oxides

Applications of functional oxides:

Microelectronics – spintronics, photonics

Data storage – RAM

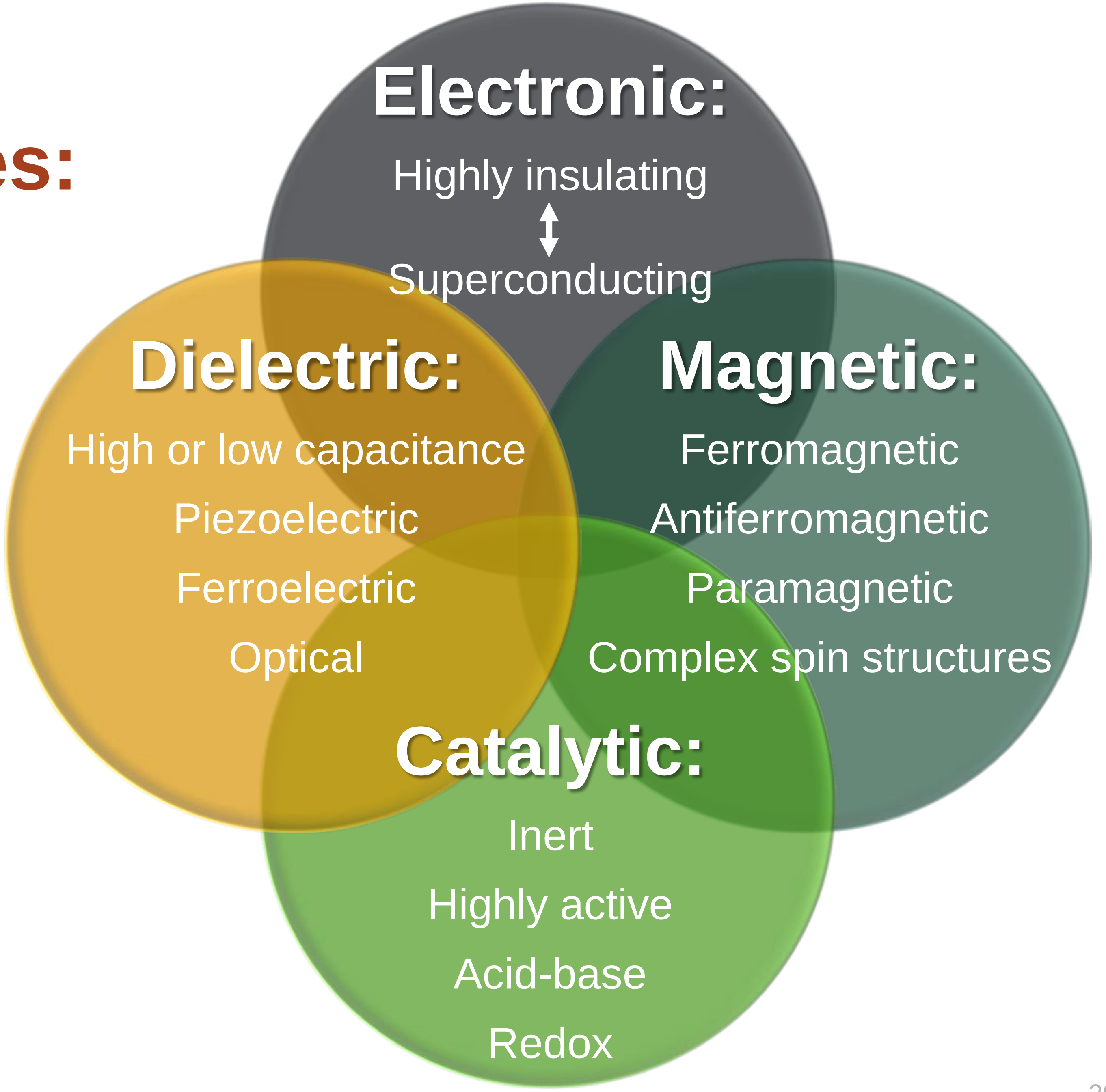
Quantum computing

Sensors – gas, chemicals

Energy generation – solar cells, fuel cells

Energy storage – ion batteries

Chemical catalysis – green chemistry



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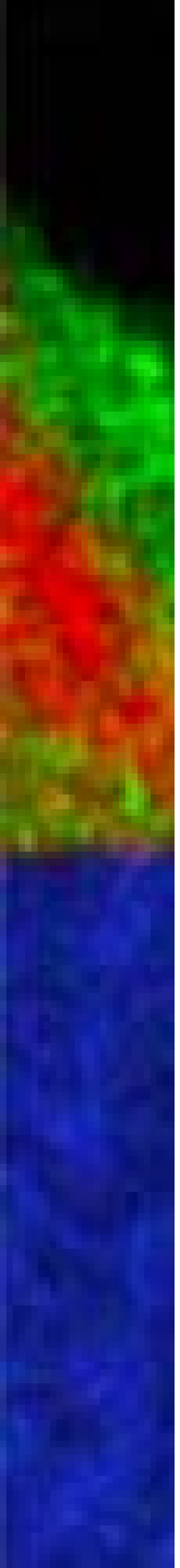
Intertwined energy and climate crisis

- **Goal: reduce or eliminate our dependence on fossil fuels**
  - Fossil fuels are a limited resource
  - Burning fossil fuels releases CO_2, causing climate change
  - Global supply chain is a national security risk
- **Solar energy is free!**
  - Harvesting it is not free – or easy – but can be very environmentally friendly
  - Generating energy near the use point leads to regional and national energy security

(Bret Hartman/Reuters)

https://www.flickr.com/photos/minagri/39828094670/



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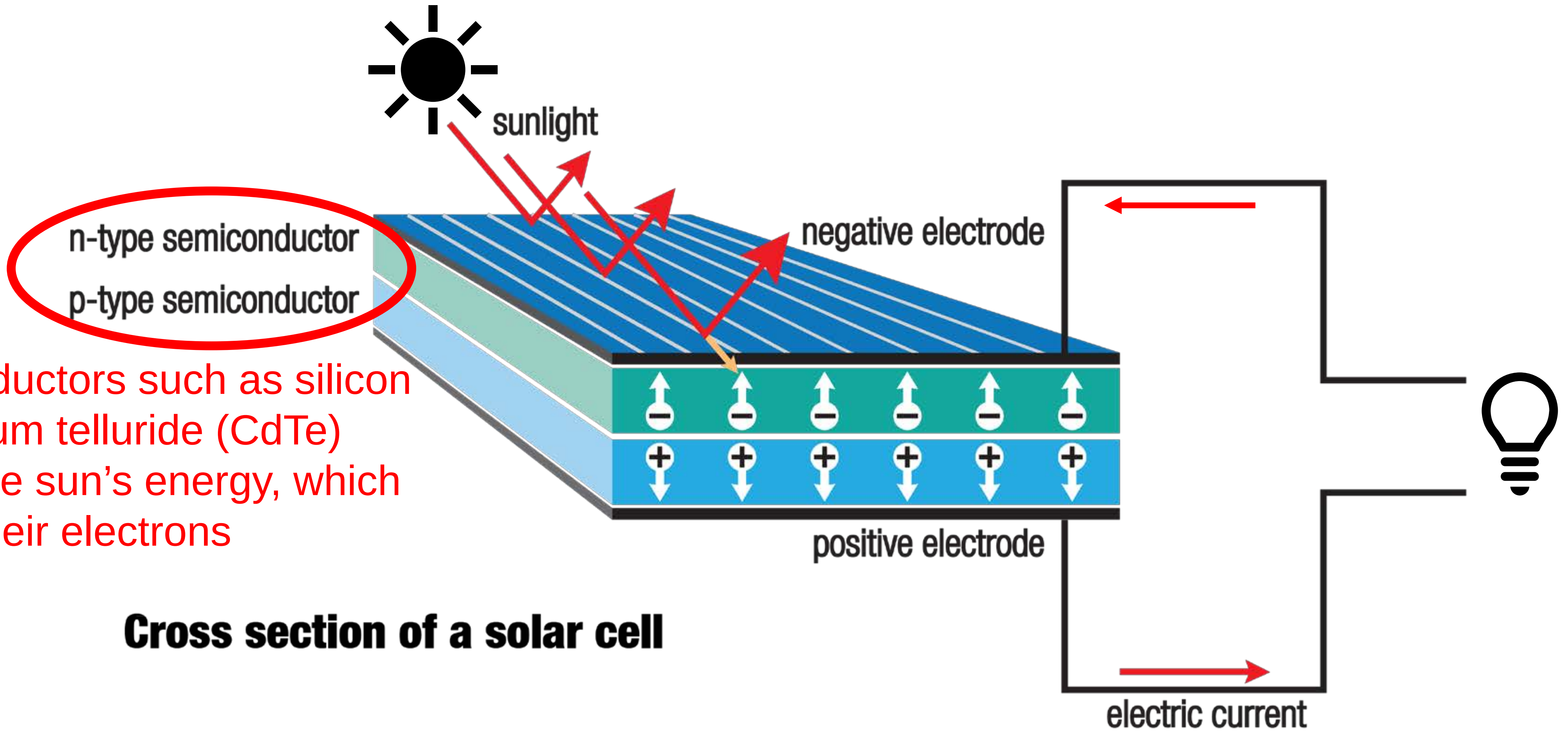
How do solar cells work?



Semiconductors such as silicon or cadmium telluride (CdTe) absorb the sun's energy, which excites their electrons

https://www.visualcapitalist.com/animation-how-solar-panels-work/

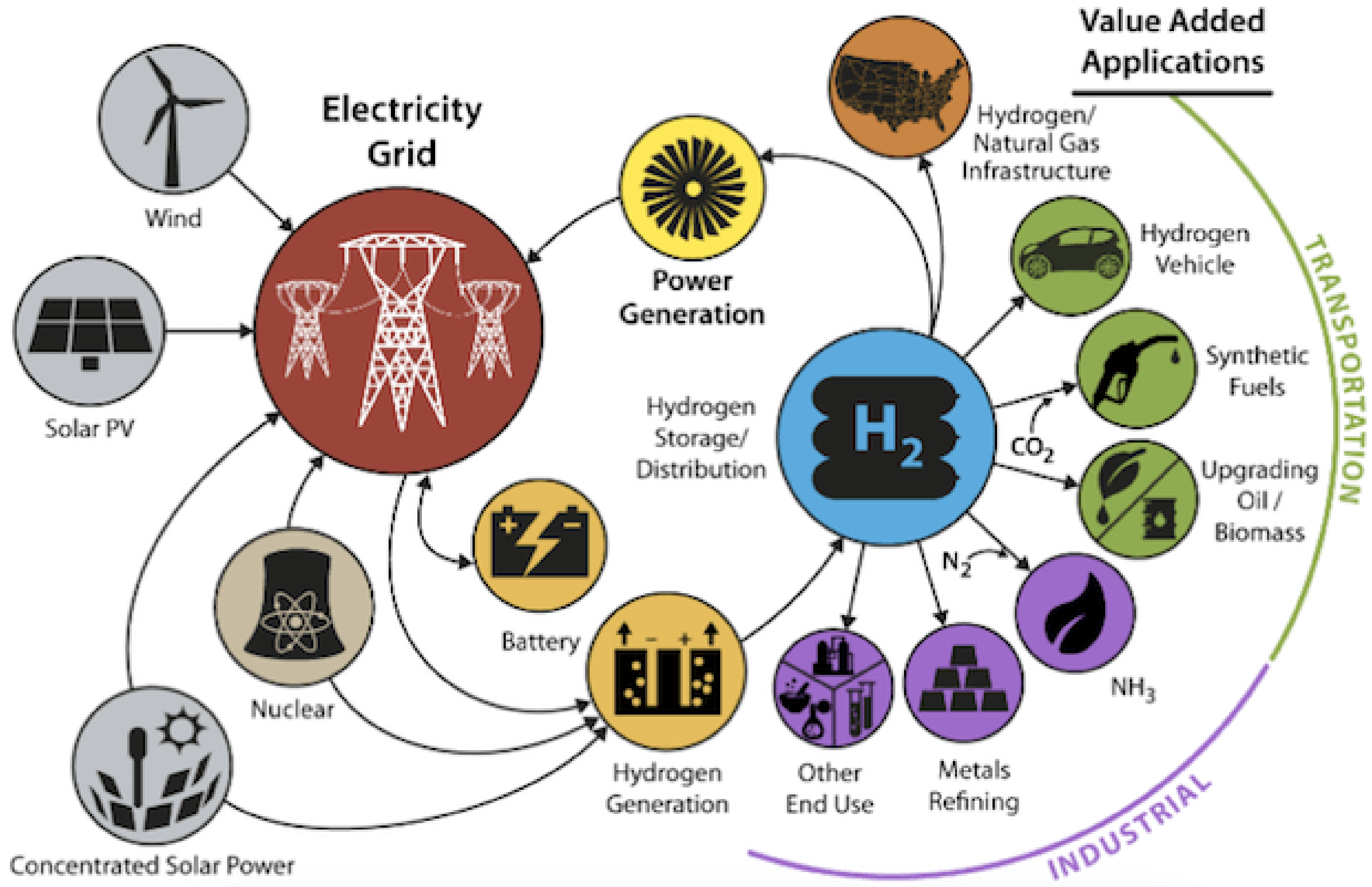
Areas for improvement:

Higher efficiency (more electricity)
Cheaper materials
Energy storage because sunlight is intermittent

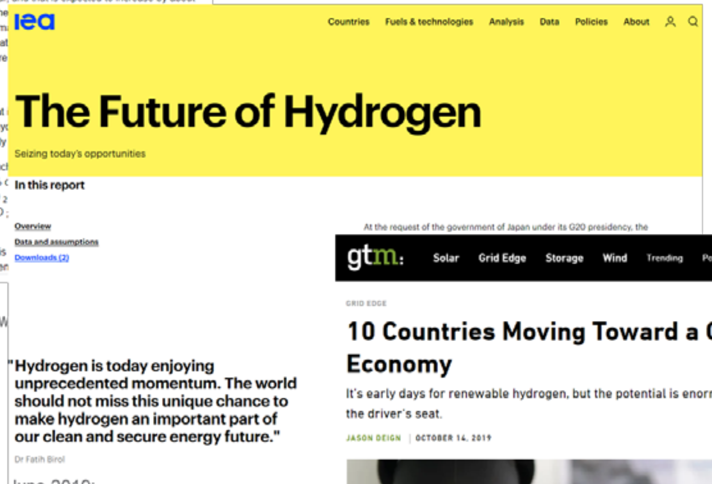
Alternative approach

Store solar energy as a **fuel**
- Energy carrier
- Portable
- Used on demand

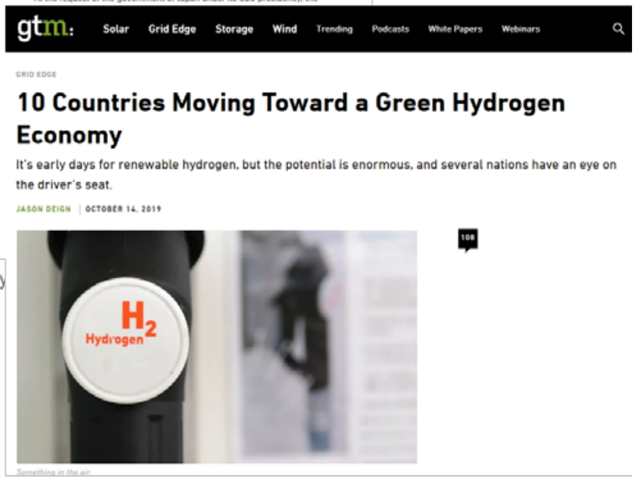
Hydrogen (H_2) is a promising fuel with a high energy density



June 2020: https://www.powermag.com/at-the-dawn-of-the-hydrogen-economy/



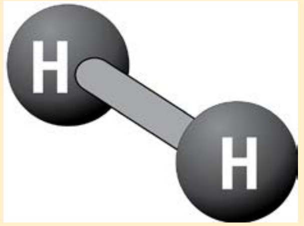
June 2019: https://www.iea.org/reports/the-future-of-hydrogen



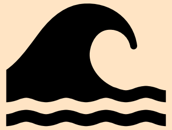
October 2019: https://www.greentechmedia.com/articles/read/10-countries-moving-towards-a-green-hydrogen-economy

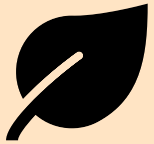
Getting to know hydrogen

The H-H chemical bond in H_2 stores a lot of energy, making H_2 an energy-dense fuel

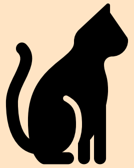


Hydrogen is literally all around us - it makes up 75% of the elemental mass of the *universe*! – but H_2 in Earth's atmosphere is only 1 part per million (ppm)



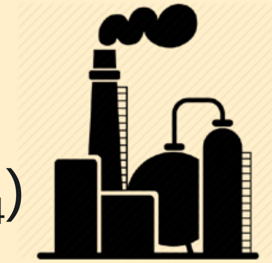


Most hydrogen on Earth is chemically bonded with other elements to make water (H_2O) and organic compounds

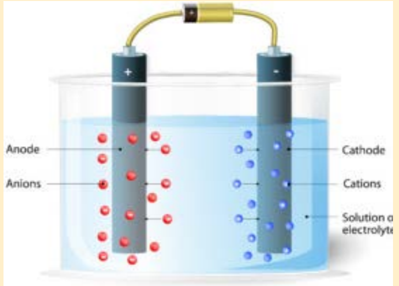


Manufacture of H_2:

Steam reforming of methane (CH_4)



Electrolysis of water

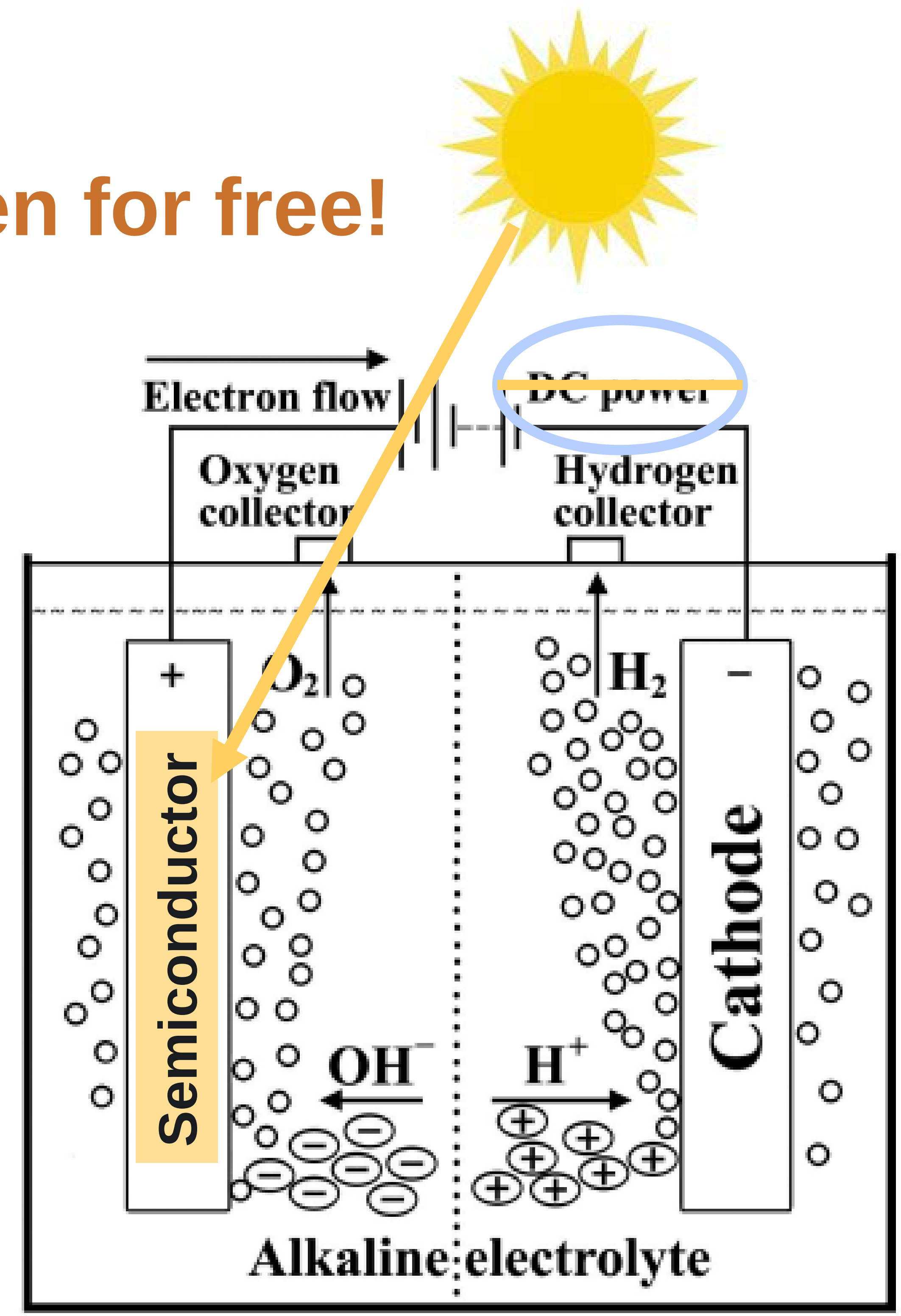


What if we could get H_2 for (almost) free with no environmental impact?

Hydrogen for free!

Electrolysis of H_2O:

- Anode and cathode (electrodes) placed in water
- Apply energy in form of voltage across electrodes
- H_2O splits and forms O_2 at anode, H_2 at cathode
- Energy-loss process: more energy is supplied (voltage) than obtained in H_2 bonds

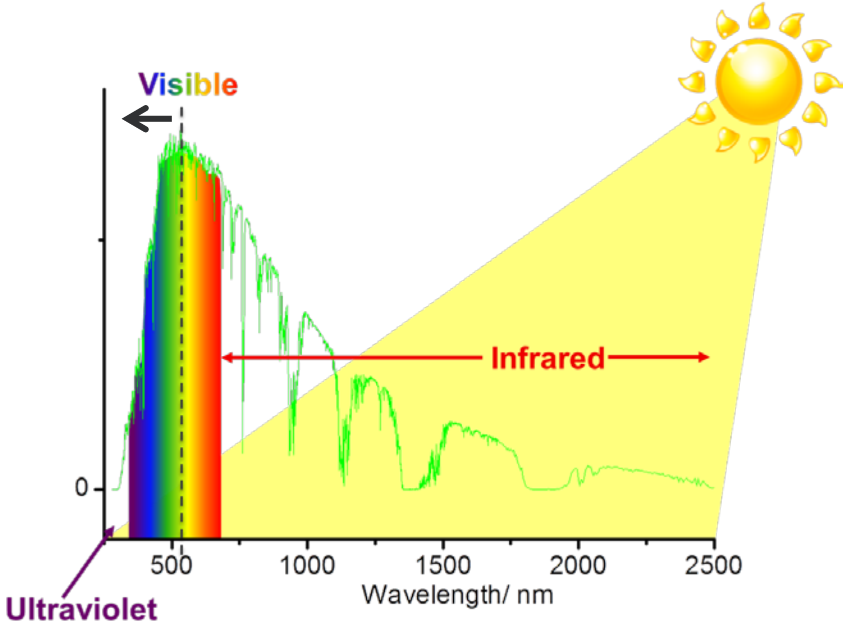


Improvement: Solar water splitting (Photoelectrolysis)

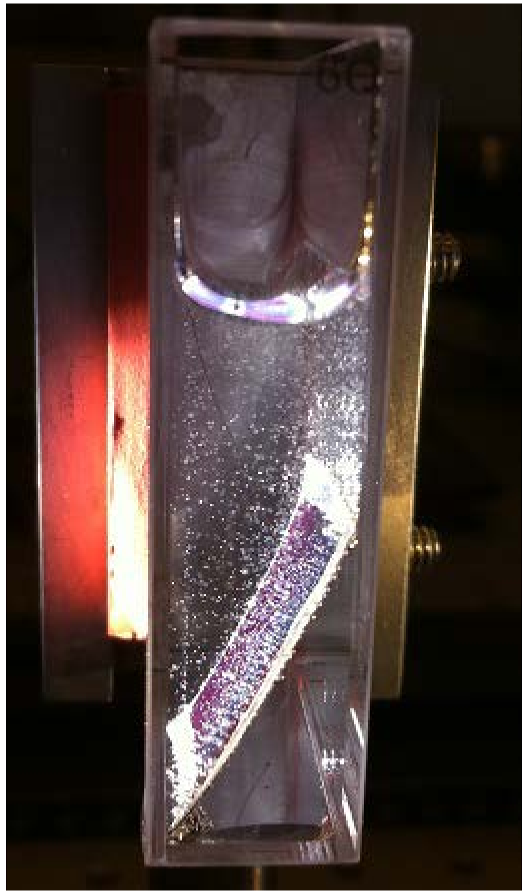
- Replace anode with semiconductor
- Replace applied voltage with sunlight
- Semiconductor anode absorbs sunlight, generating electrons and holes that react to split H_2O
- Energy-efficient process!

https://www.researchgate.net/figure/Basic-scheme-of-a-water-electrolysis-system_fig3_262617097

What are the properties of a good photocatalyst?



Narrow bandgap to absorb most of the solar spectrum
Ideal: ~1.5 eV



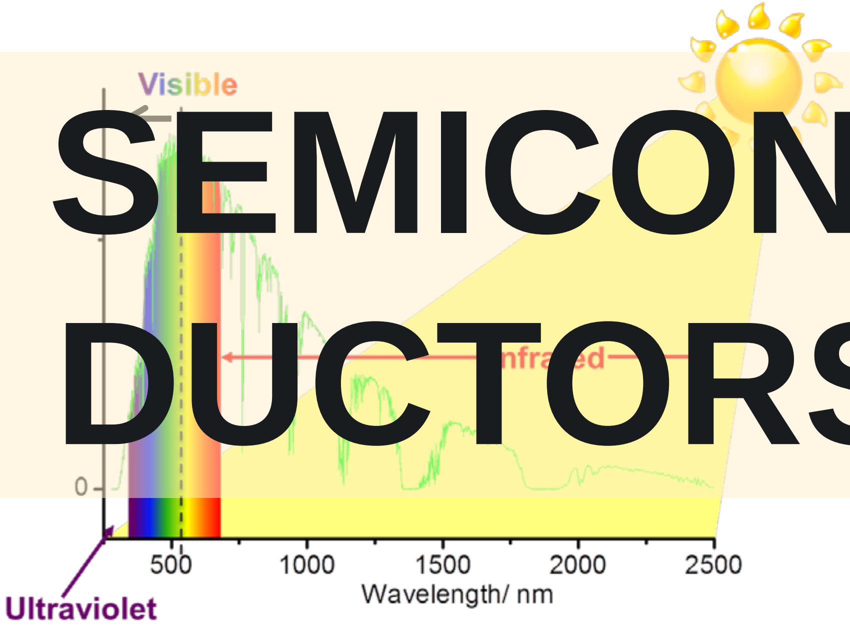
Stable in aqueous (water) environments

Earth-abundant elements for economical scalability

Preferably non-toxic

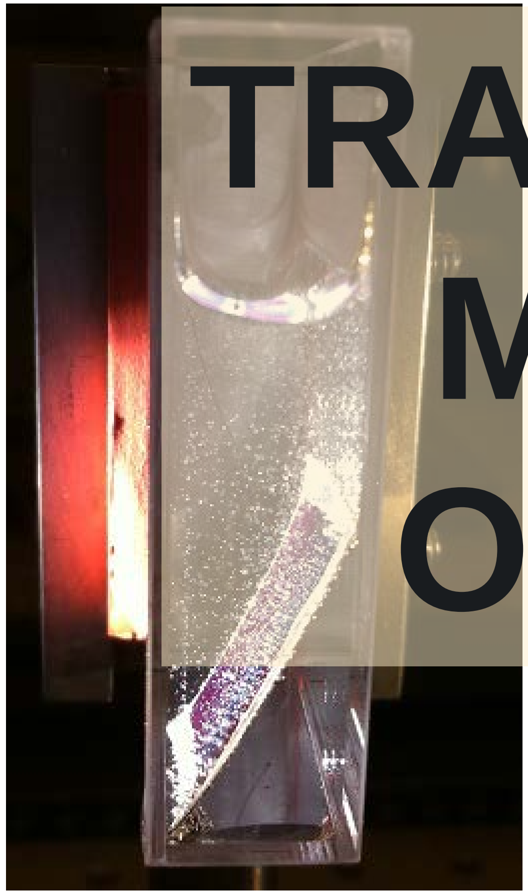
What are the properties of a good photocatalyst?

SEMICON-DUCTORS



Narrow bandgap to absorb most of the solar spectrum
Ideal: ~1.5 eV

TRANSITION METAL OXIDES



Stable in aqueous (water) environments

Earth-abundant elements for economical scalability

Preferably non-toxic

Let’s use rust!

Hematite (Fe_2O_3) is an appealing photocatalyst:

Narrow bandgap (2.1 eV) to absorb much of the solar spectrum

Stable in aqueous environments

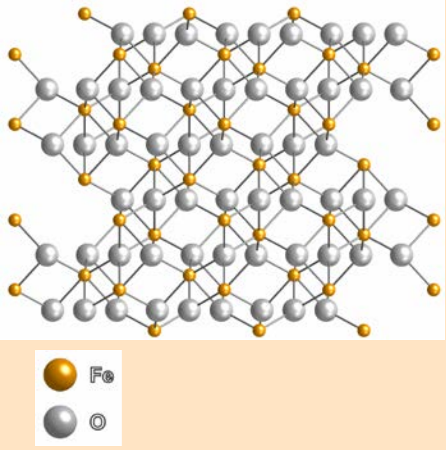
Non-toxic: important in biology

Earth abundance: 4th most abundant element in the crust

Tuning the properties of hematite thin films

Drawbacks to hematite (Fe_2O_3) for solar water splitting:

Hematite crystal structure



Too electrically insulating

Does not adsorb all of the solar spectrum

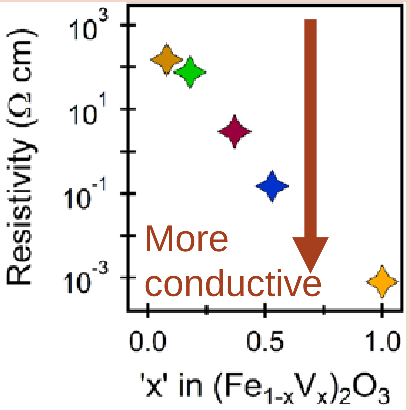
Does not efficiently turn sunlight into electrons

Improve the electrical conductivity of hematite:

Replace some Fe ions with Ti or V

Ti or V donates one extra electron to the material

These extra electrons increase the electrical conductivity

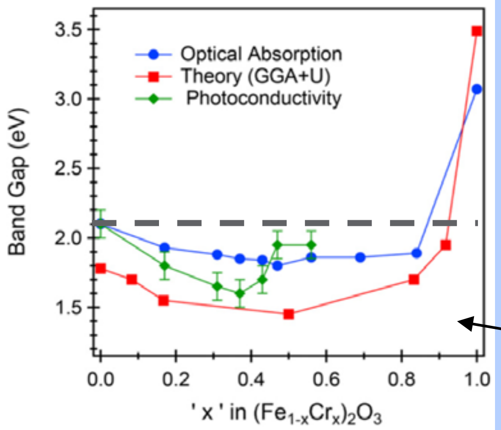


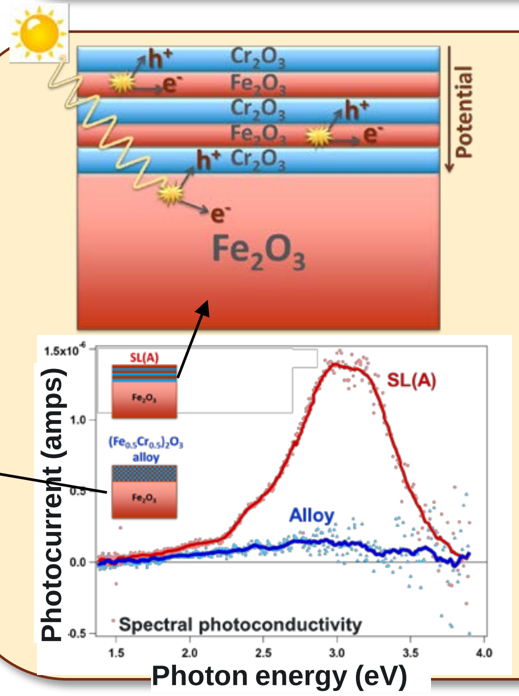
Improve the range of sunlight absorption:

Replace some Fe ions with Cr

Addition of Cr ions narrows the bandgap below Fe_2O_3 or Cr_2O_3

Narrower bandgap can absorb more of the solar spectrum



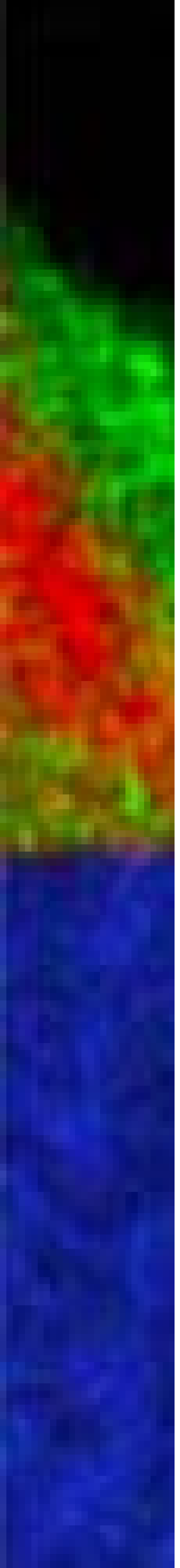


Improve the sunlight-to-electrons efficiency

Need to separate negative electrons from positive holes

Separate with voltage

Grow Fe_2O_3/Cr_2O_3 layers with a built-in voltage (potential)



Beyond Fe_2O_3 and summary

Beyond Fe_2O_3: Fe_2CrO_4

Based on magnetite (Fe_3O_4) lattice

Magnetite has high (metallic) conductivity

Introducing Cr lowers the conductivity; Fe_2CrO_4 is semiconducting

Improved efficiency to turn sunlight into electrons

Electrical conductivity:

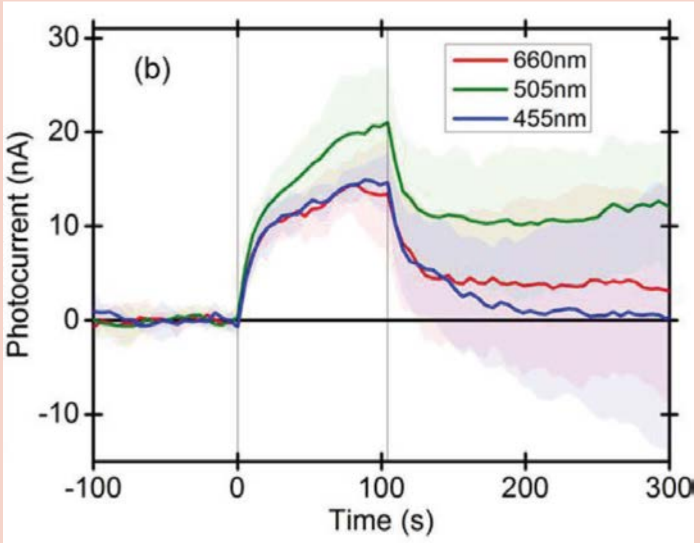
Fe_3O_4: metallic

↓

Fe_2CrO_4: semiconducting

↓

$FeCr_2O_4$: insulating



Summary of water splitting with iron oxides

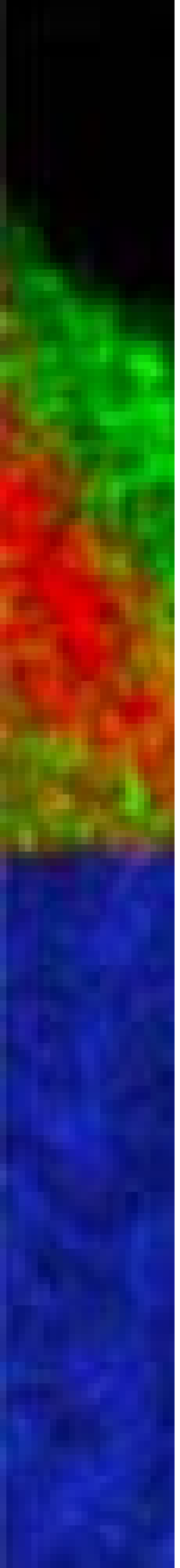
We have engineered the properties of Fe_2O_3 by doping with Ti, V, Cr

Gained fundamental insight into the effect of these dopants on Fe_2O_3

Explored the semiconducting spinel Fe_2CrO_4

These fundamental studies are the goal of our research

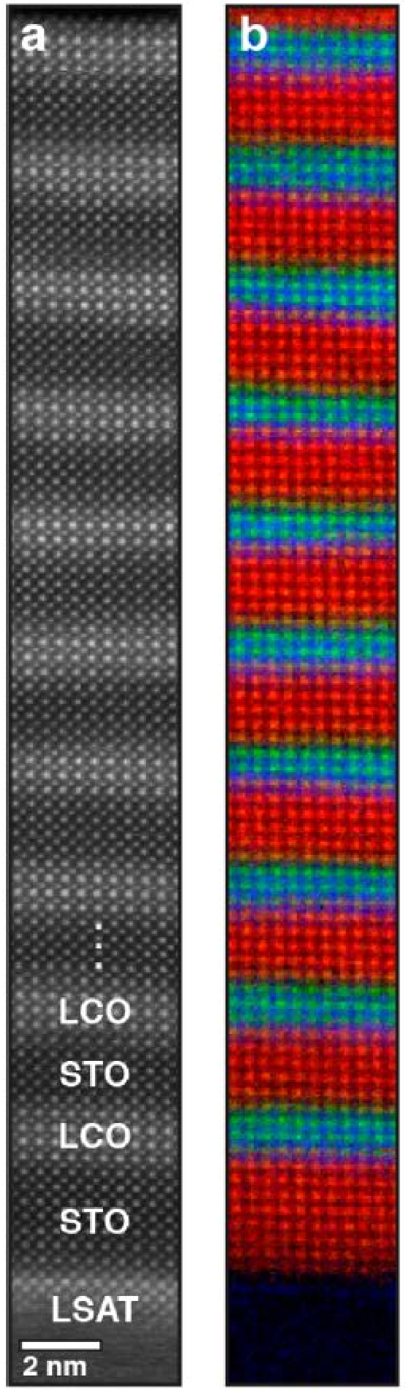
Others are using these results in applications of iron oxides



Outlook for functional oxides

- Modern technology is built on materials science – understanding the chemistry and physics of materials
- The underlying principles that result in the wide range of properties of oxides are beginning to be understood systematically
  - Epitaxial thin films are key to these systematic studies
- The next frontier: emergent properties at interfaces between oxides

http://images.lipy.com/bang



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https://www.surveymonkey.com/r/PNNL090820

Join us for our next webinar

Solving the World's Biggest Problems on the Smallest Scale

Presented by: Heather Olson
Tuesday, October 13, 2020
7:00 pm
ZOOM

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THANK YOU!

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