Design and Growth of Novel Materials

Paul C. Canfield

Distinguished Professor,

Department of Physics

Senior Physicist, Ames Laboratory

Iowa State University

Physics 590B
Spring 2019

By the end of this lecture I want you to know where single crystalline samples come from and the effort that goes into discovering and optimizing new materials and properties.

The reason for this is simple. Many physicists have a poor idea of what is involved. This means that they can be lazy or sloppy when it comes to understanding the importance (as well as the limitations) of single crystalline samples.

Let's start with a story….

Once upon a time, there was a nice single crystal

THAT GOT DROPPED AND BROKEN BY A HURRIED STUDENT

There was a great anger in the land.

Do you have *ANY* idea where single crystal samples come from????!!!!

Thanks to David Mandrus for archival photo….

Yes…

Said the little student….

Yes…

Single crystal come from

FedEx
Express

Yes…

<u>NO….I want you to know the proper reply.</u>

This class will draw heavily from these three papers

Proceedings of the 2nd Annual European School in Material Science Ljubljana, Slovenia May 2007.

CHAPTER 2

GROWTH PAPER ONE

SOLUTION GROWTH OF INTERMETALLIC SINGLE CRYSTALS: A BEGINNER'S GUIDE.

Paul C. Canfield

PHILOSOPHICAL MAGAZINE B, 1992, VOL. 65, NO. 6, 1117–1123

Growth of single crystals from metallic fluxes

By P. C. CANFIELD and Z. FISK

Los Alamos National Laboratory,
Los Alamos, New Mexico 87545, USA

GROWTH PAPER TWO

Journal of Crystal Growth 225 (2001) 155–161

High-temperature solution growth of intermetallic single crystals and quasicrystals

Paul C. Canfield*, Ian R. Fisher

GROWTH PAPER THREE

New Materials development / single crystal growth is often done with very modest equipment

The spaces are small, and there can be a delivery room-like atmosphere. Crystal growth, like birth, can be messy. It is a fantastic, exciting, and addictive experience, *(spontaneous symmetry breaking at its best)*.

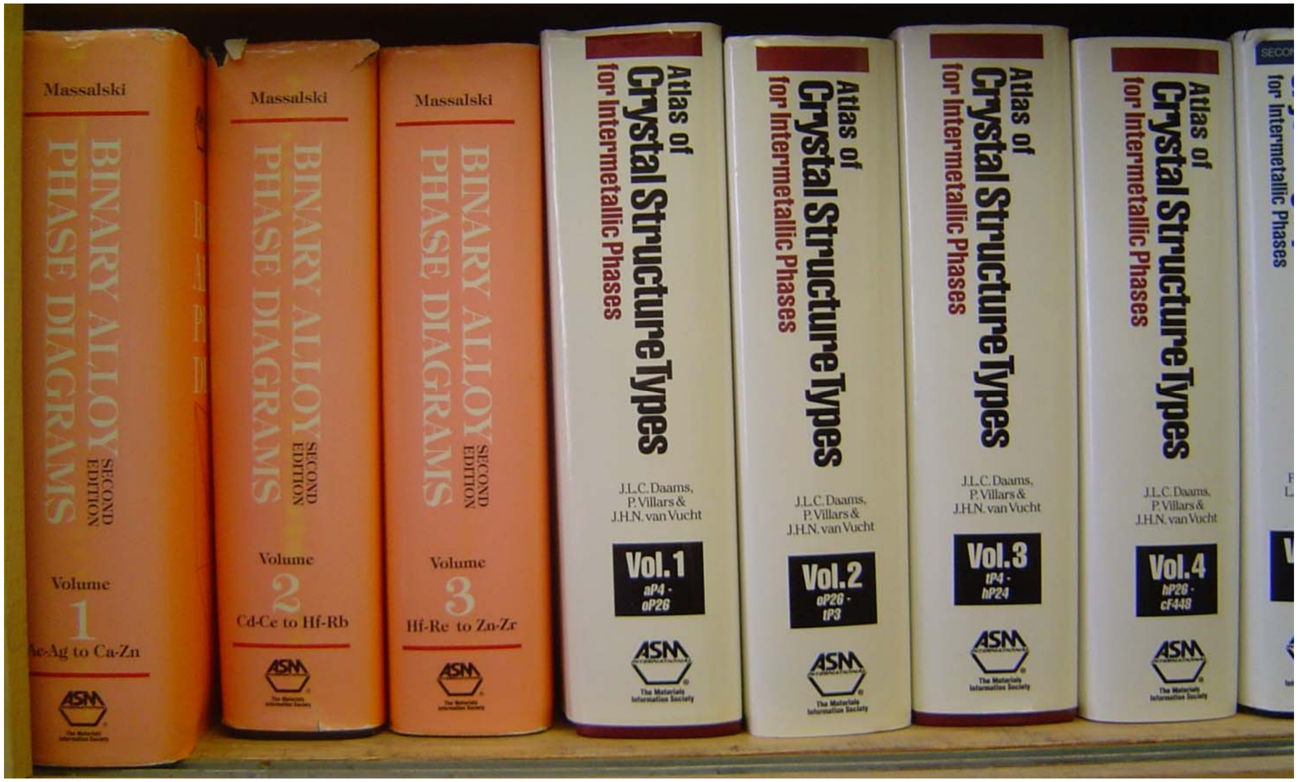
Let's see how this works

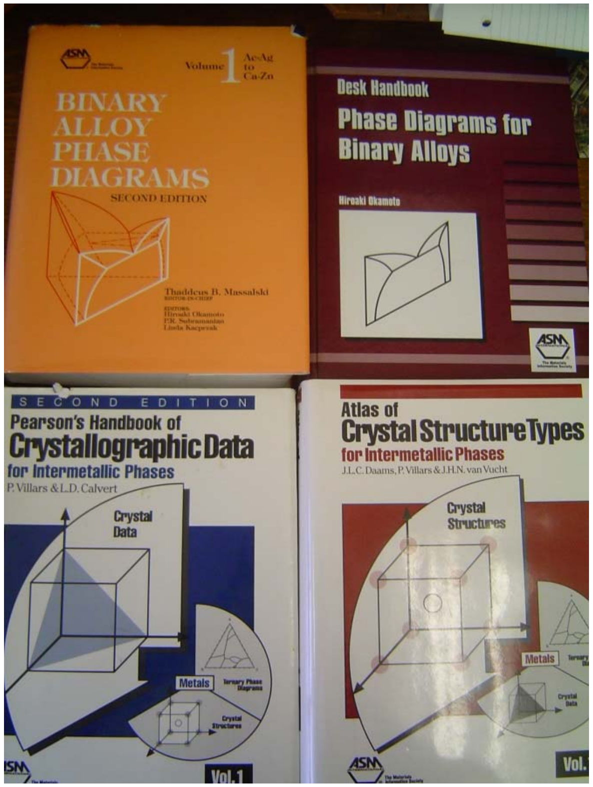
First you need an idea….

This is often the tricky part of research….

Next, you should check some phase diagrams to see what might work….

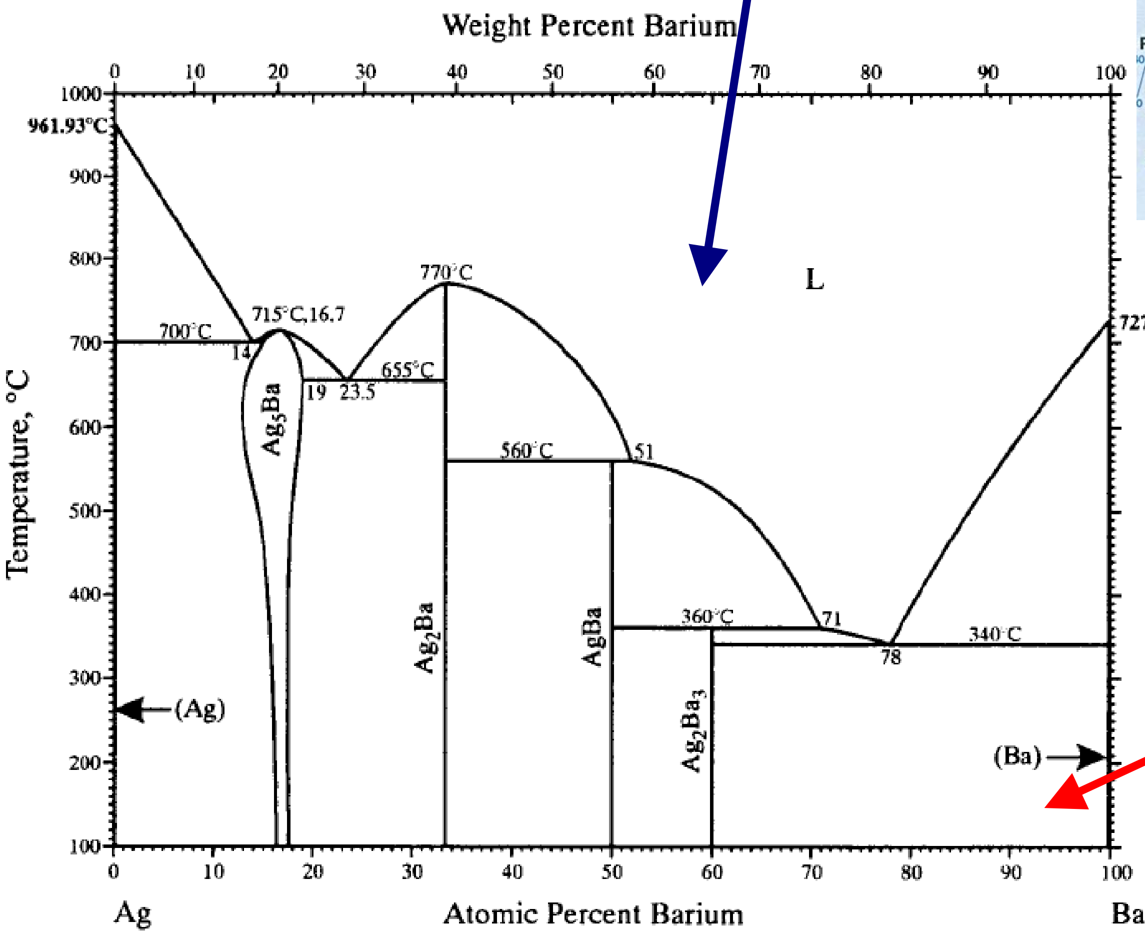
Most of the diagrams I will show come from these books.





Binary phase diagrams are fundamentally like maps: they show the extent of liquid and solid.

Liquids



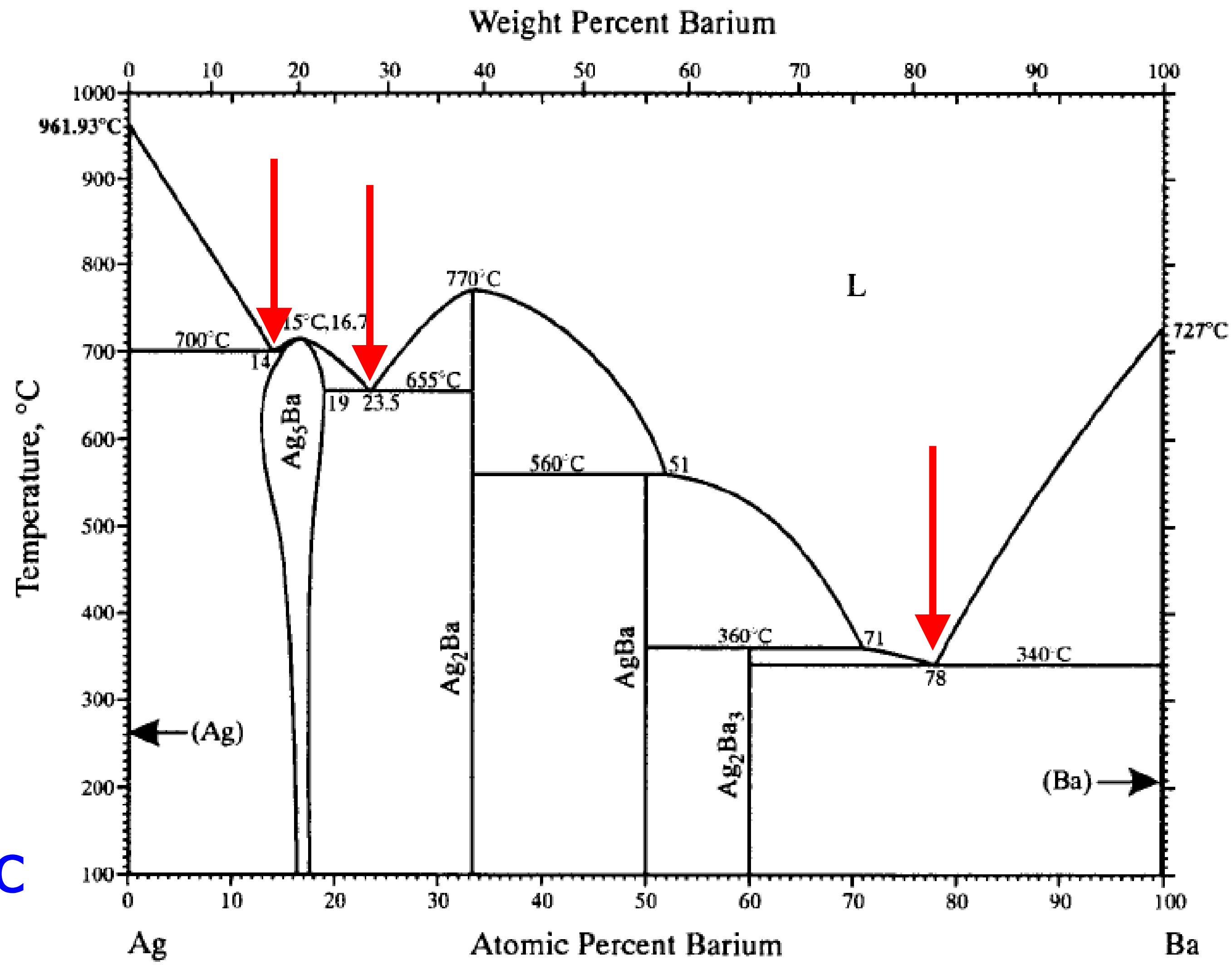
Solids

Let’s develop a little vocabulary

Eutectic points: minima in liquid regions

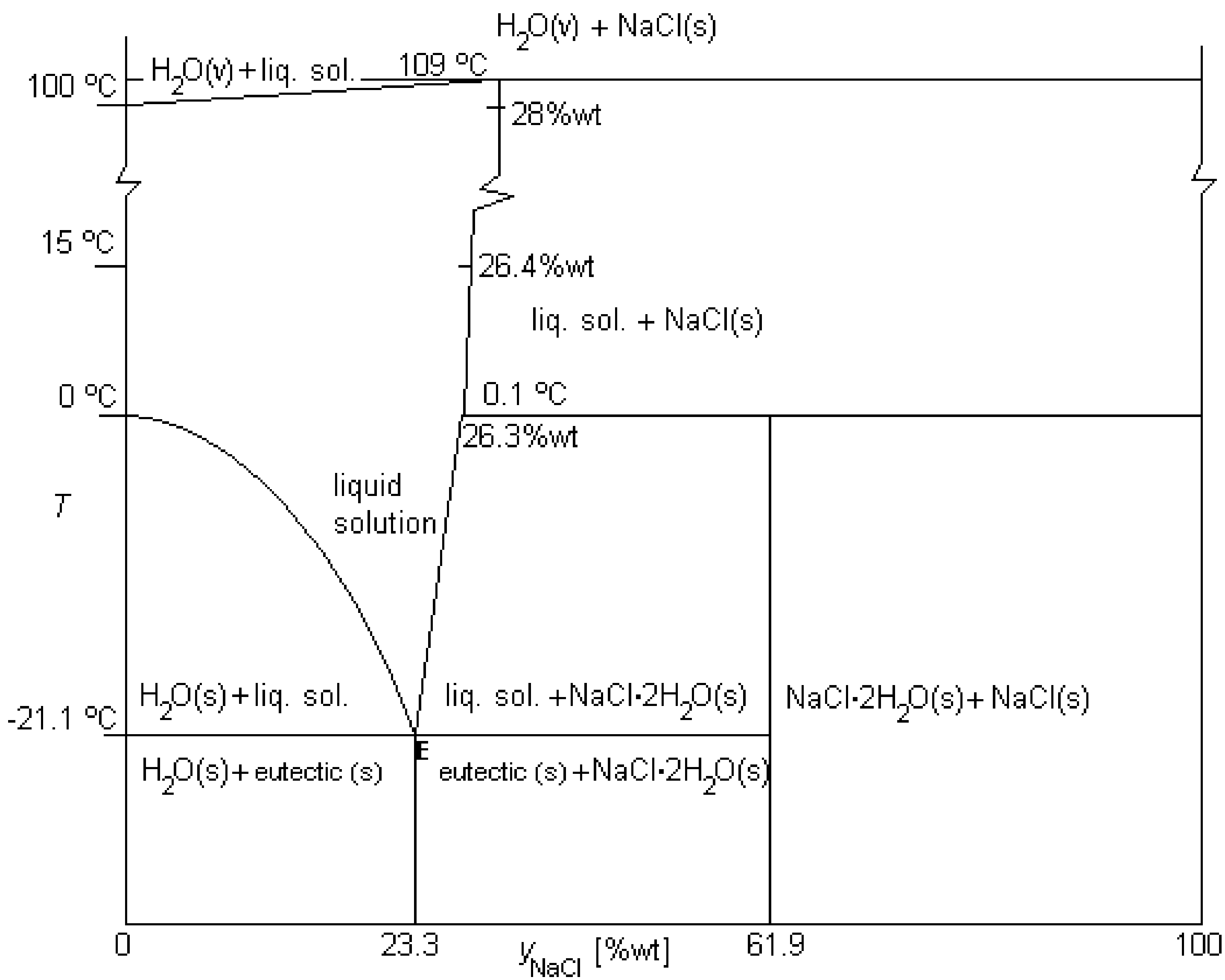
Solution growth requires readily accessible liquid regions.

Often these are found in eutectic valleys



Eutectic points: minima in liquid regions

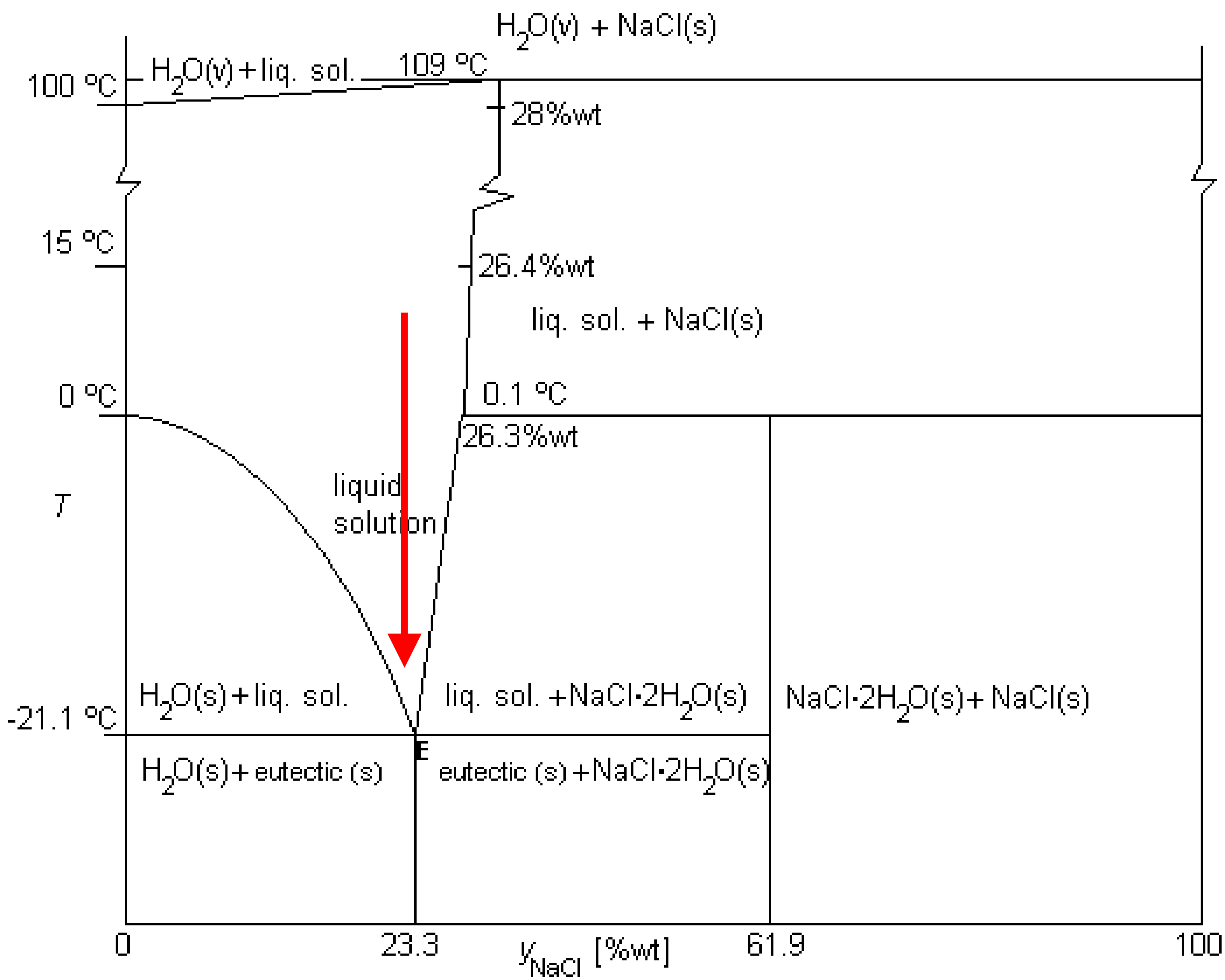
Here is a familiar one associated with melting ice with salt, or lower freezing point of sea water.



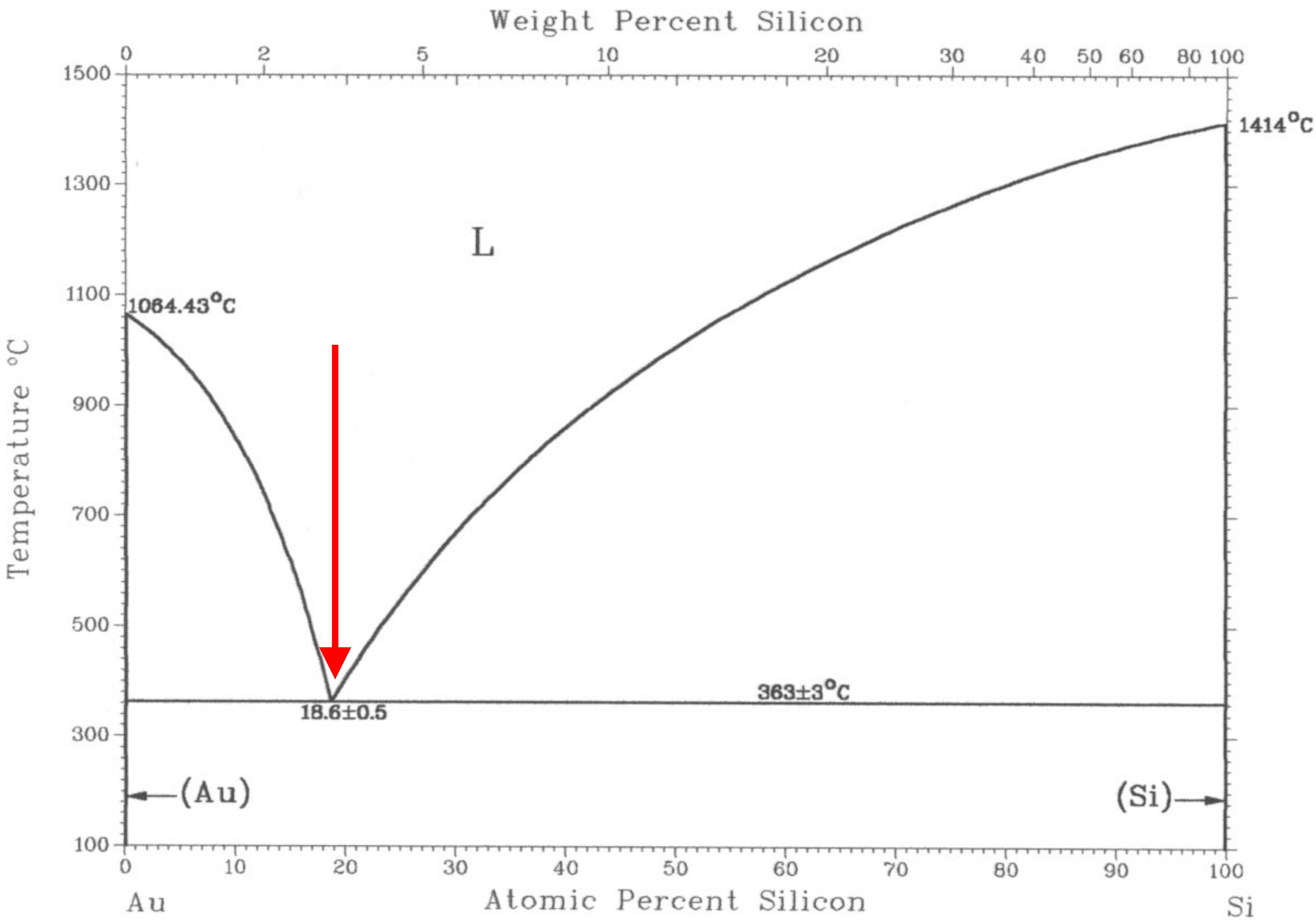
Eutectic points: minima in liquid regions

Here is a familiar one associated with melting ice with salt, or lower freezing point of sea water.

At 23.3 weight percent NaCl, H_2O melts / solidifies just below -21 ºC

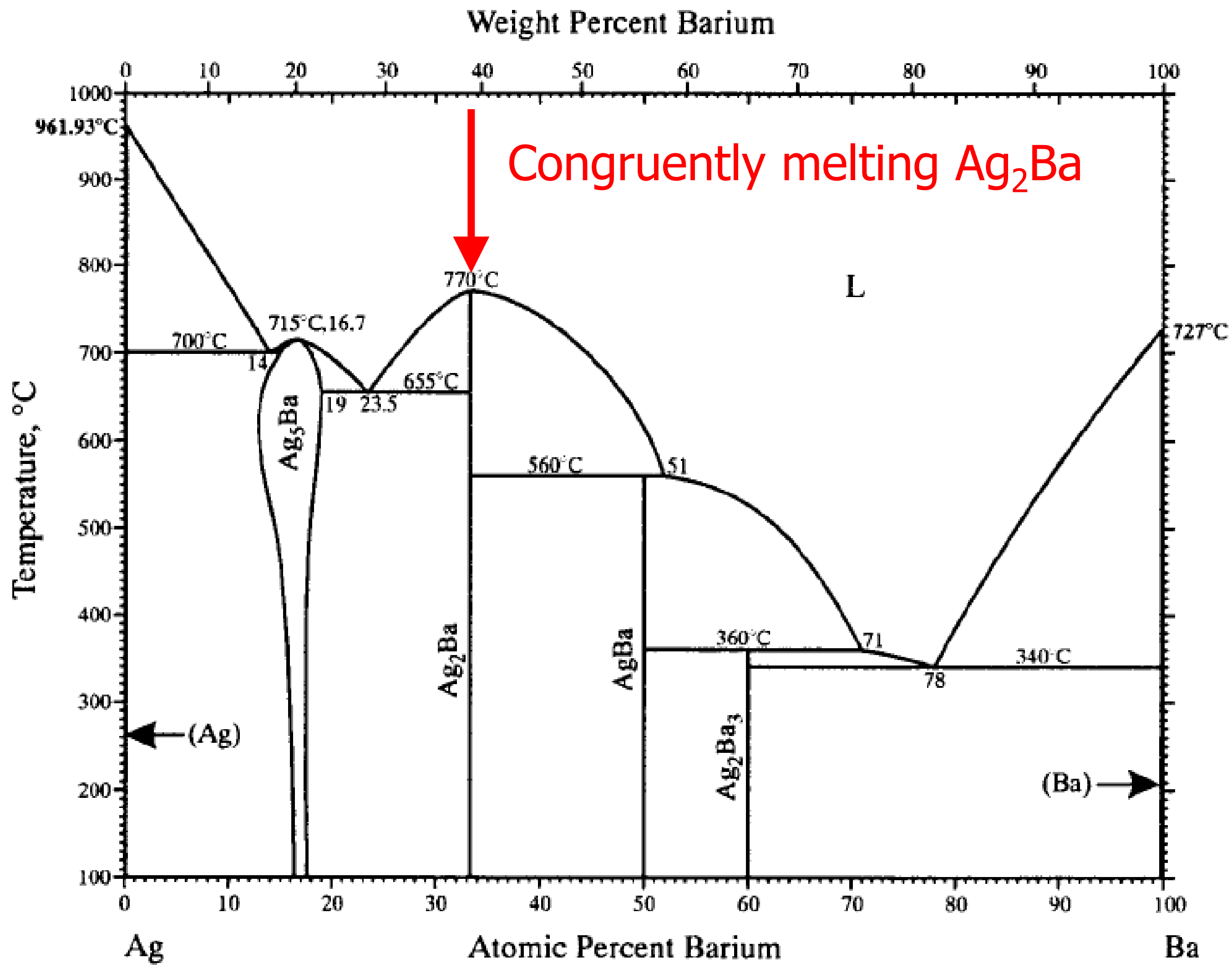


Deep Eutectics offer tempting liquid regions for growth



Congruently melting compounds transform from a homogeneous solid to a homogeneous liquid at melting point.

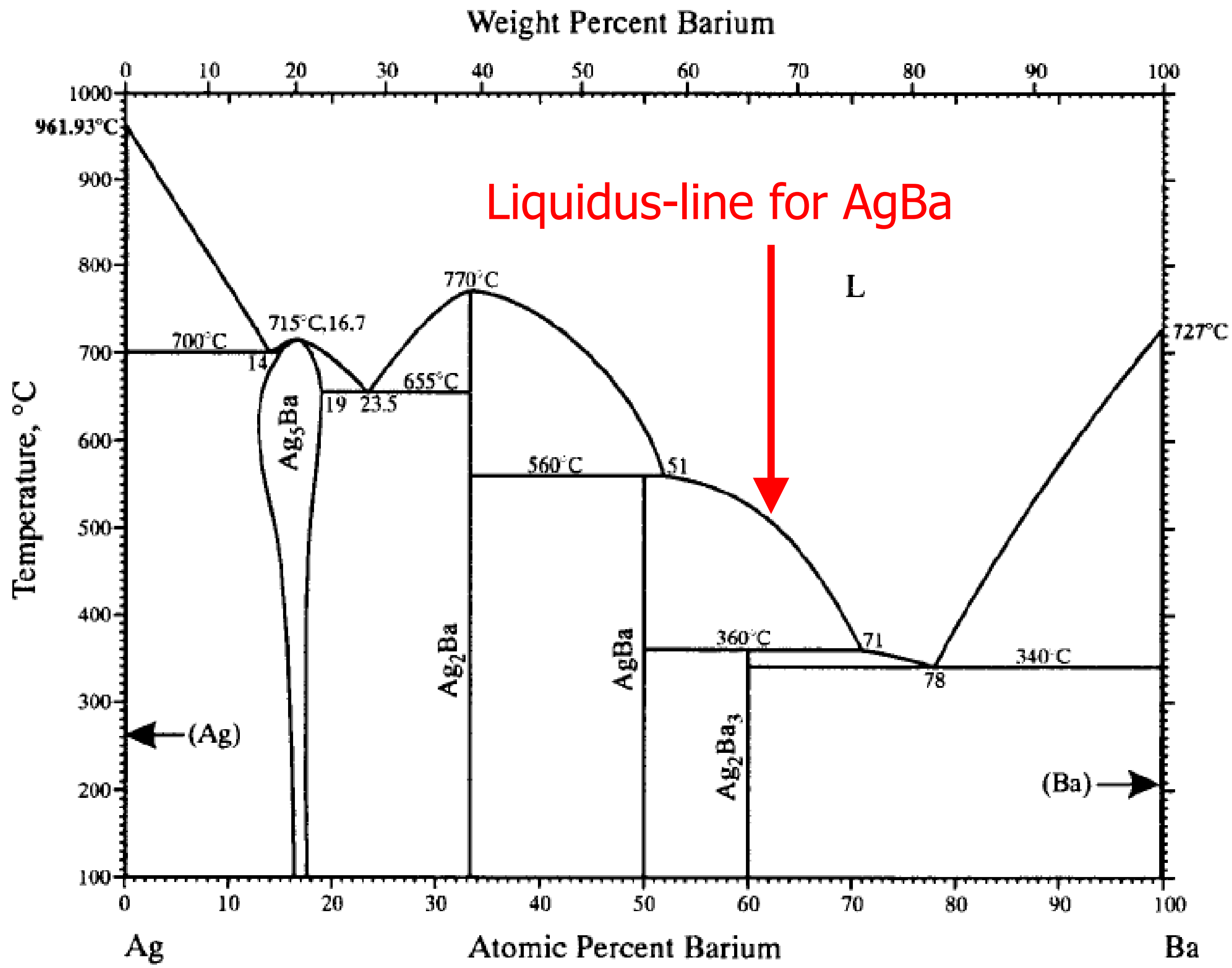
Congruently melting compounds can be made by a wide variety of growth techniques that simply melt and solidify samples of fixed composition.



Liquidus-line

As we cool through the liquid, we ultimately cool enough to hit the liquidus-line for AgBa. At this temperature AgBa starts crystallizing and the remaining liquid becomes more Ba rich. The sample is no longer homogeneous and instead contains a solid of one stoichiometry and a liquid of another, changing stoichiometry.

KEY to solution / flux growth!

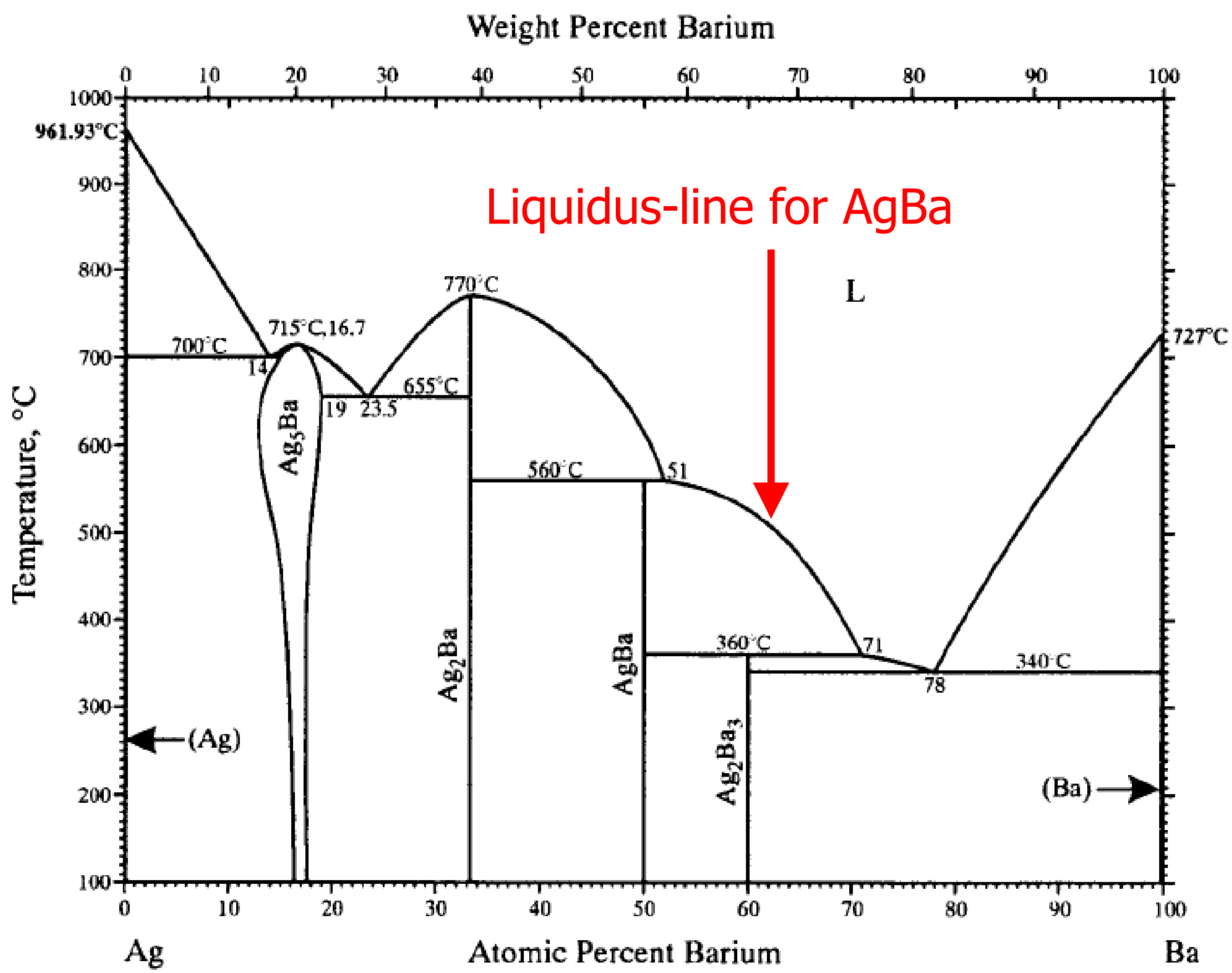


Liquidus-line

KEY to solution / flux growth!

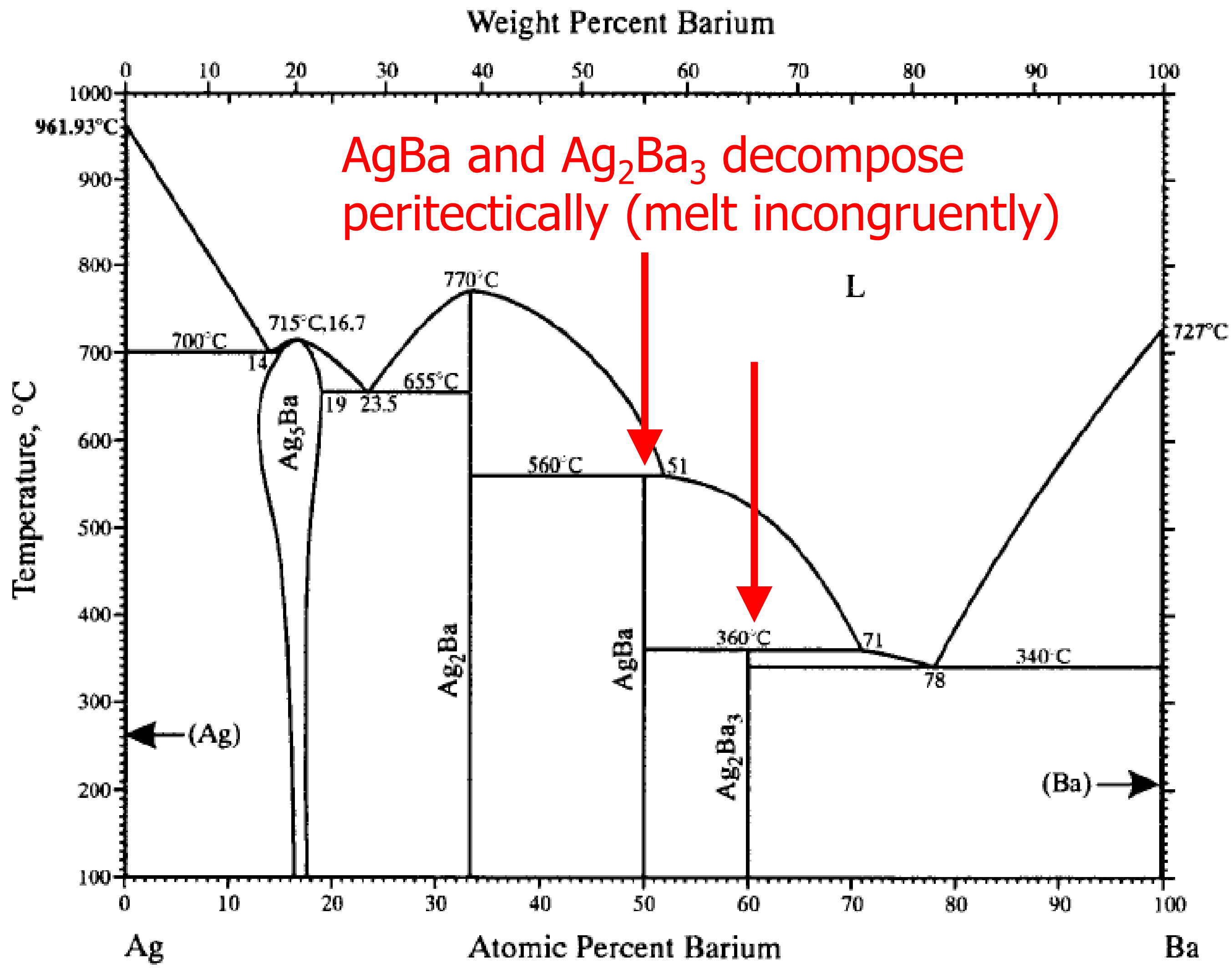
Once we cool low enough to hit the liquidus line, the system enters a TWO PHASE REGION. The liquidus line tells us the composition of the remaining liquid, which changes as we form solid phases.

More on this in few slides.



Incongruently melting compounds undergo a peritectic decomposition into a mixed solid and liquid phase, only to form a homogeneous liquid at higher temperatures.

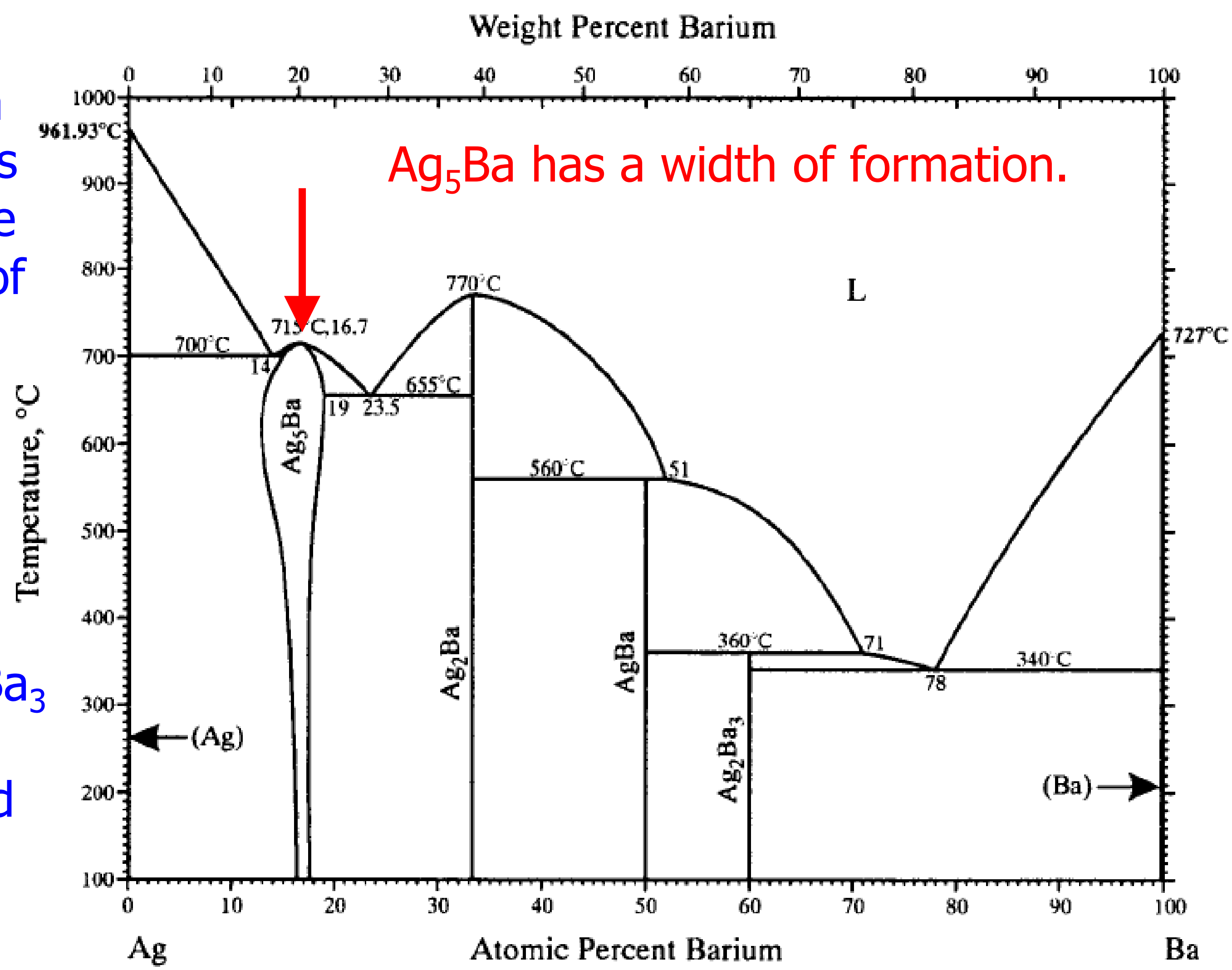
If you cool a liquid with composition AgBa, it will first form Ag_2Ba as the remaining liquid becomes more Ba rich and only forms AgBa below the peritectic temperature of 560 C.



Widths of formation and line compounds.

Ag_5Ba can form with a variety of compositions and even have a single crystal with a spread of stoichiometries.

Ag_2Ba, $AgBa$ and Ag_2Ba_3 are show to have no width of formation and are called “line” compounds.



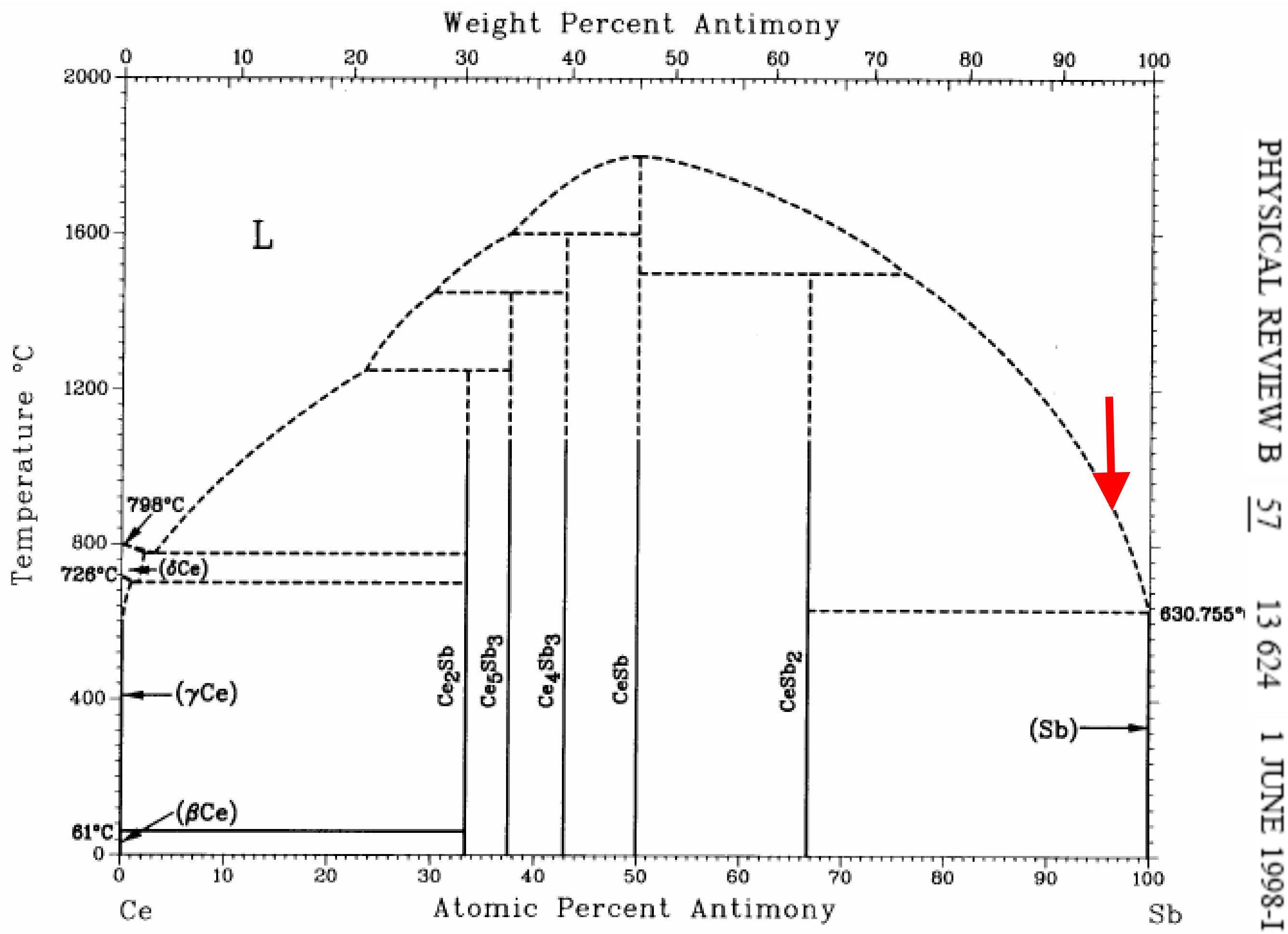
Crystal growth of $CeSb_2$?

This is all nice in theory….But how do we REALLY DO THIS???

WITH OUR HAND AND BODIES NOT SOME MENTAL EXERCISE

$CeSb_2$ is incongruently melting at a relatively high temperature. An attempt to cool a melt of $CeSb_2$ would end up with a mixed phase and lots of mess (high vapor pressures).

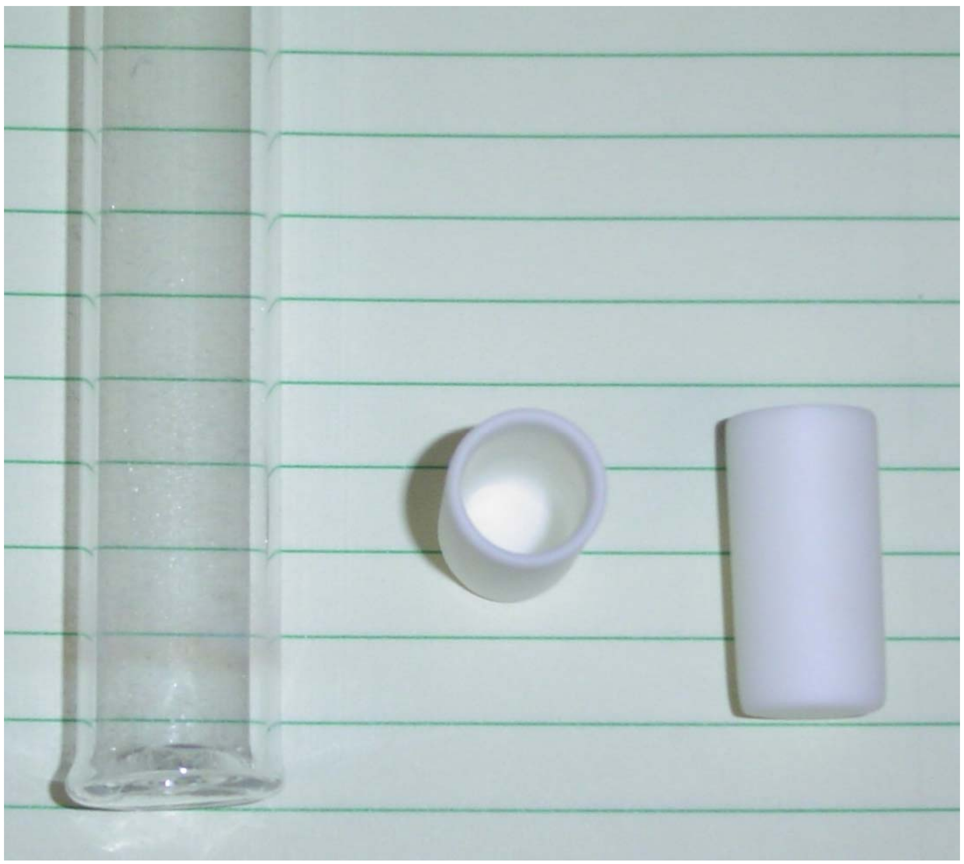
On the other hand there is a very open line of primary solidification. Grow $CeSb_2$ out of a “self flux” of excess Sb.

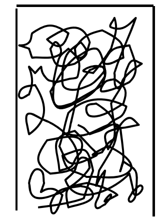


As a rule of thumb, if we are below ~12 % R we can use Al_2O_3 crucibles

We need to seal the crucible in a silica tube to contain and protect the growth

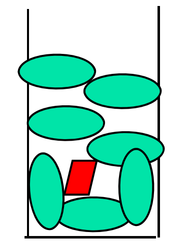
Shown below are two 2 ml crucibles and a snugly fitting silica tube



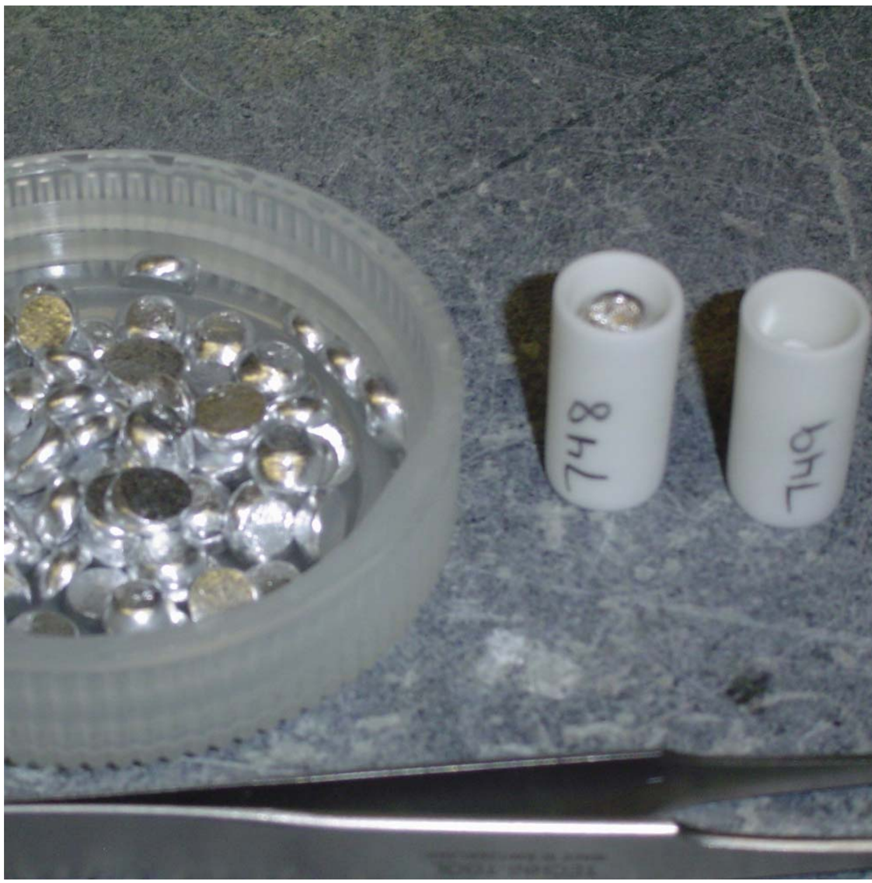


Place the Ce and Sb into the growth crucibles

Put quartz wool into the catch crucible



Place the growth crucibles and catch crucibles into the silica tubing



Place quartz wool on top of the crucibles

Use the H_2-O_2 torch to neck down the silica

Evacuate the silica and seal off the ampoules

Clean off any finger prints, grease, etc.

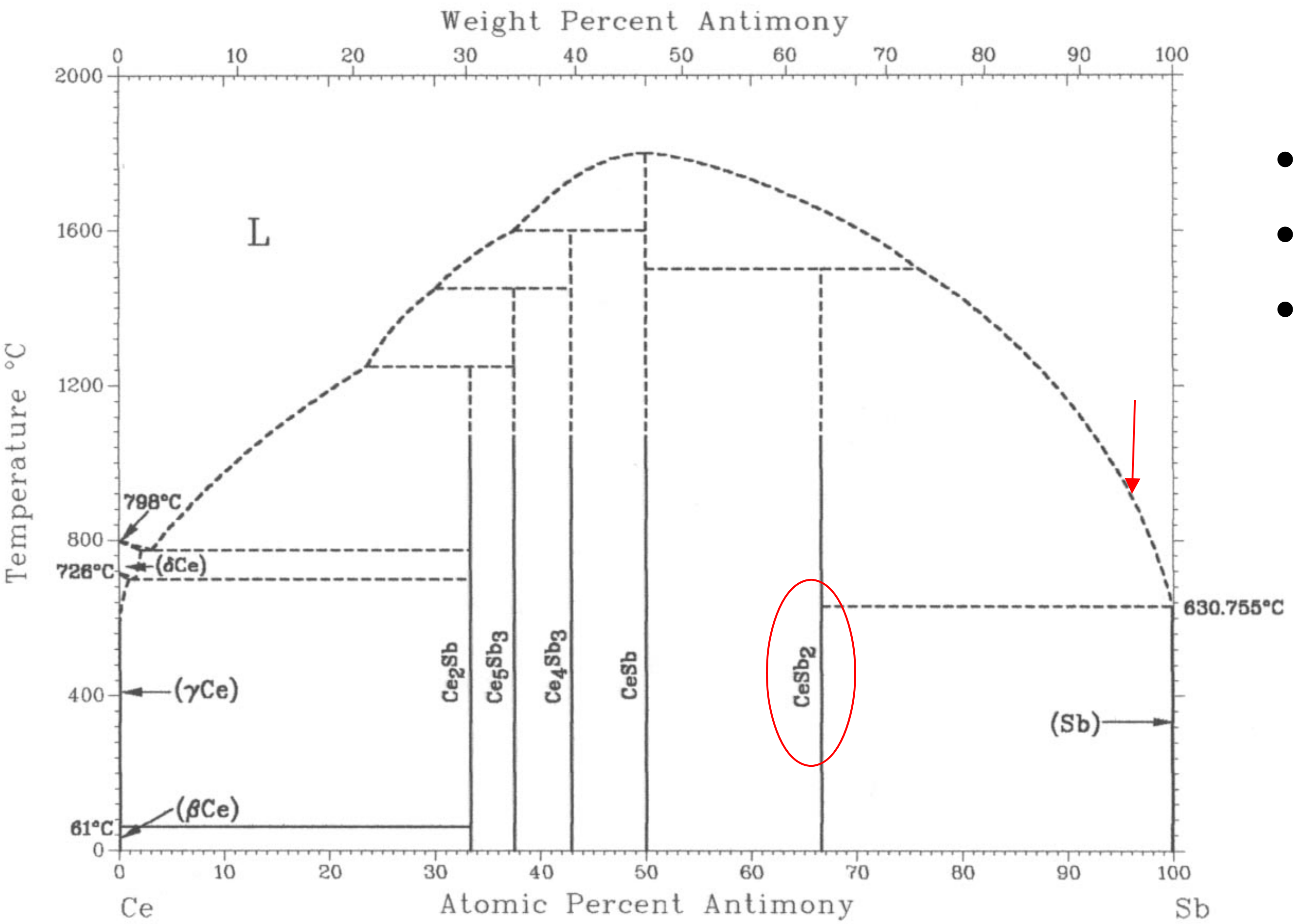
Place ampoules into furnace

Program the temperature – time profile and let the thermodynamics take place

When growth is done, pour off the excess liquid.
(a = 10-1000 g is better than a = 1 g = 9.8 m/s^2)

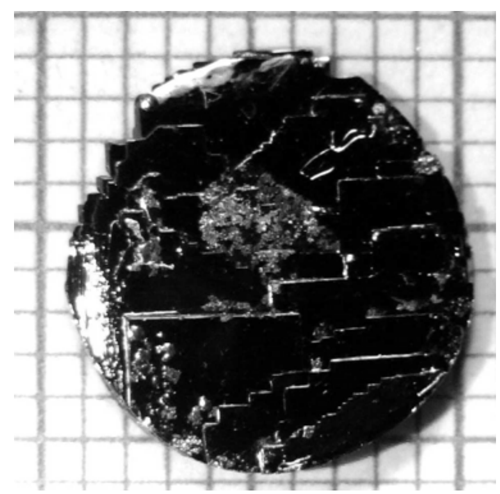
Flux growth (slow cooling of a melt)

Basic idea: slow cool into 2-phase region



eg: $CeSb_2$ / Sb

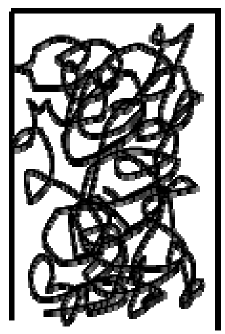
- self flux
- $Ce_{0.05}Sb_{0.95}$
- 1190 °C → 700 °C

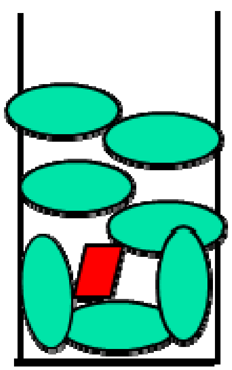


PHILOSOPHICAL MAGAZINE, 2016
VOL. 96, NO. 1, 84–92
http://dx.doi.org/10.1080/14786435.2015.1122248

Use of frit-disc crucibles for routine and exploratory solution growth of single crystalline samples

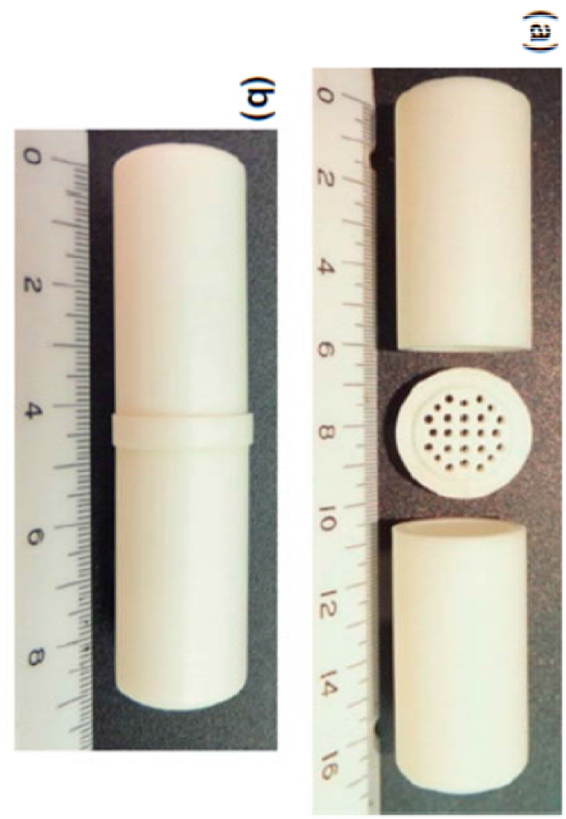
Paul C. Canfield, Tai Kong, Udhara S. Kaluarachchi and Na Hyun Jo

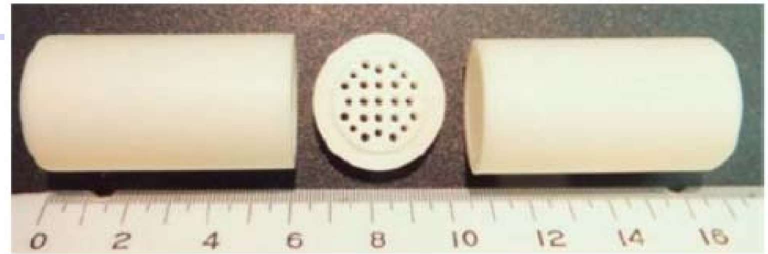




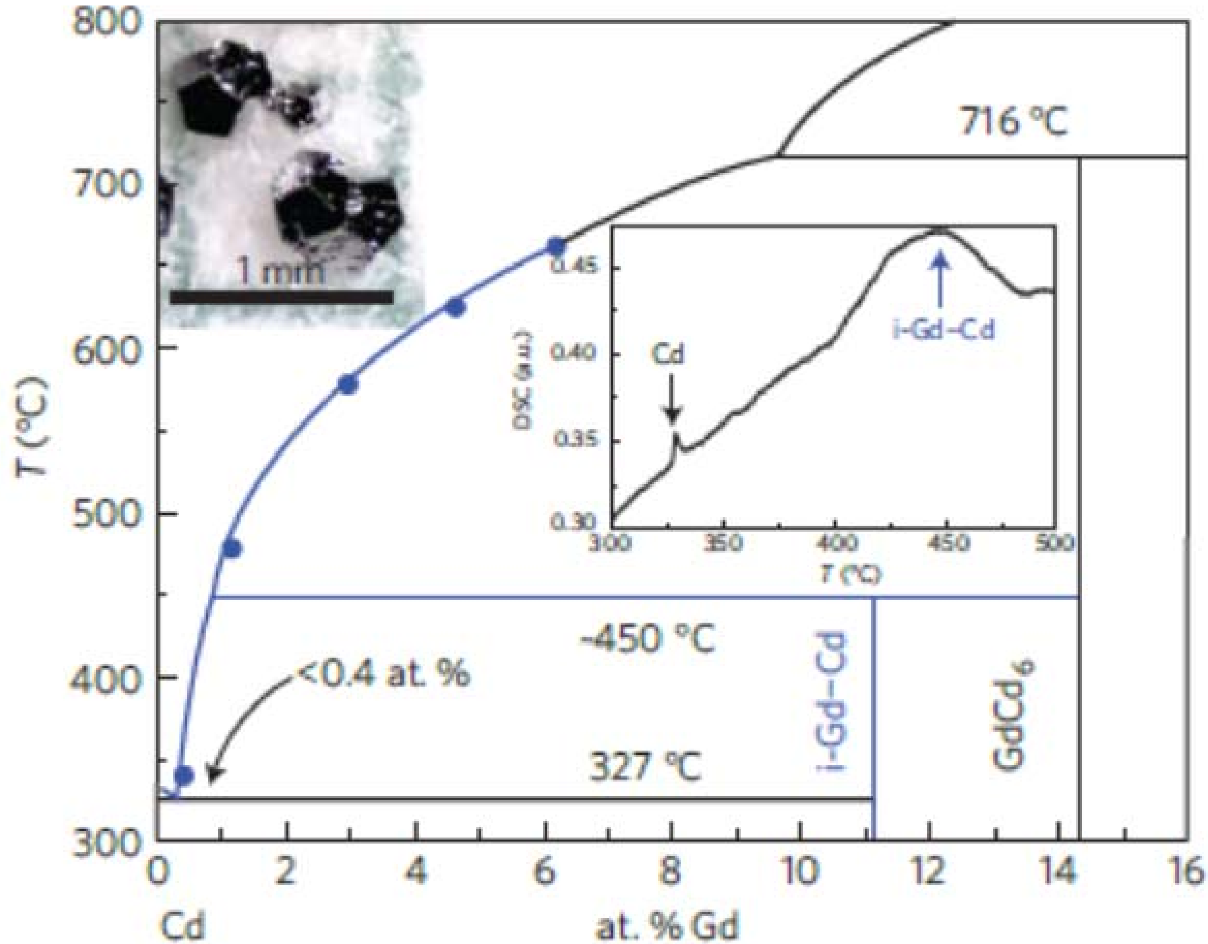
Instead of using silica wool as a filtering agent, we recently developed a frit-disc filtering mechanism and were able to integrate it with the alumina crucibles.

This allows for clean separation of the solids and liquid phases during the decanting process.





Clean recovery of both growth side as well as spin side materials allow for weighing and reuse. This allows for fractionation of the growth as well as phase diagram determination (at least in some cases).



LETTERS

PUBLISHED ONLINE: 9 JUNE 2013 | DOI: 10.1038/NMAT3672

nature materials

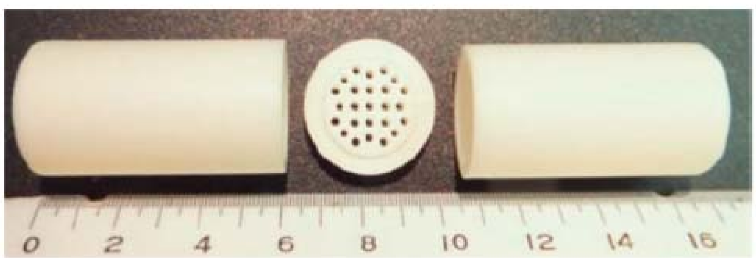
PHILOSOPHICAL MAGAZINE, 2016
VOL. 96, NO. 1, 84–92
http://dx.doi.org/10.1080/14786435.2015.1122248

Use of frit-disc crucibles for routine and exploratory solution growth of single crystalline samples

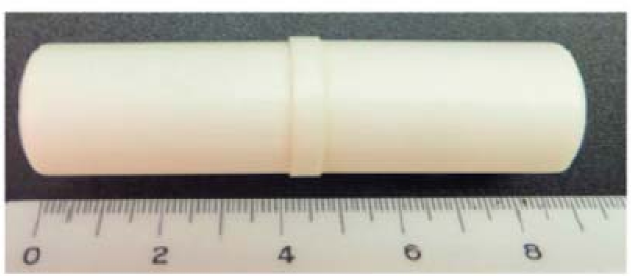
Paul C. Canfield, Tai Kong, Udhara S. Kaluarachchi and Na Hyun Jo

These three piece sets are now available in a variety of sizes

(a)

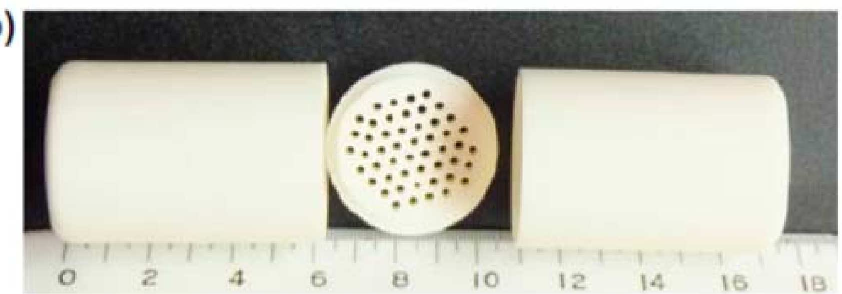


(b)



(a)

(b)



PHILOSOPHICAL MAGAZINE, 2016
VOL. 96, NO. 1, 84–92
http://dx.doi.org/10.1080/14786435.2015.1122248

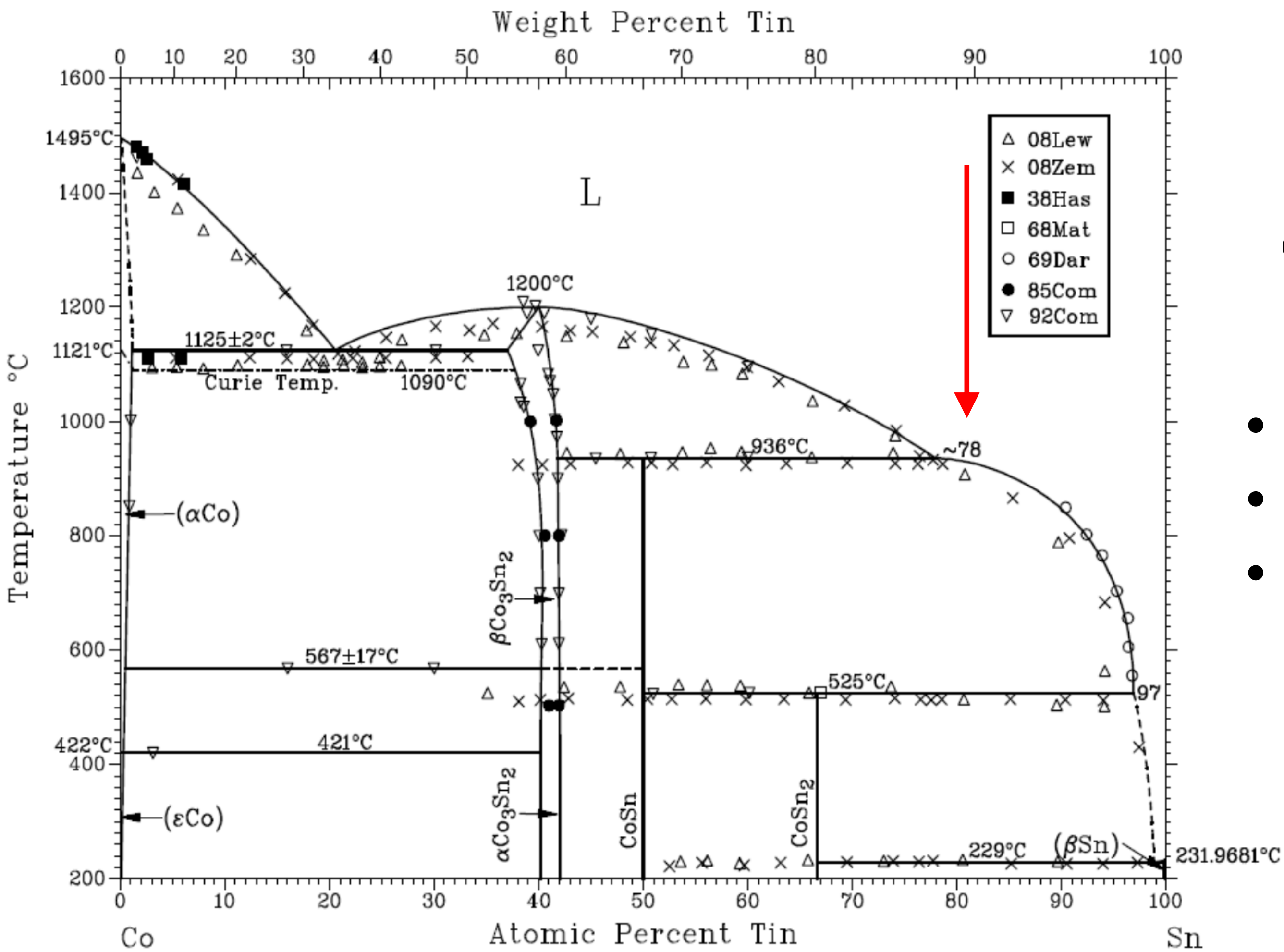
Use of frit-disc crucibles for routine and exploratory solution growth of single crystalline samples

Paul C. Canfield, Tai Kong, Udhara S. Kaluarachchi and Na Hyun Jo

2^{nd} example, let's see how to grow CoSn.

It is deeply peritectic, but it has a large exposed liquidus line.

We can cool a binary melt with more than 78% Sn and grow CoSn.



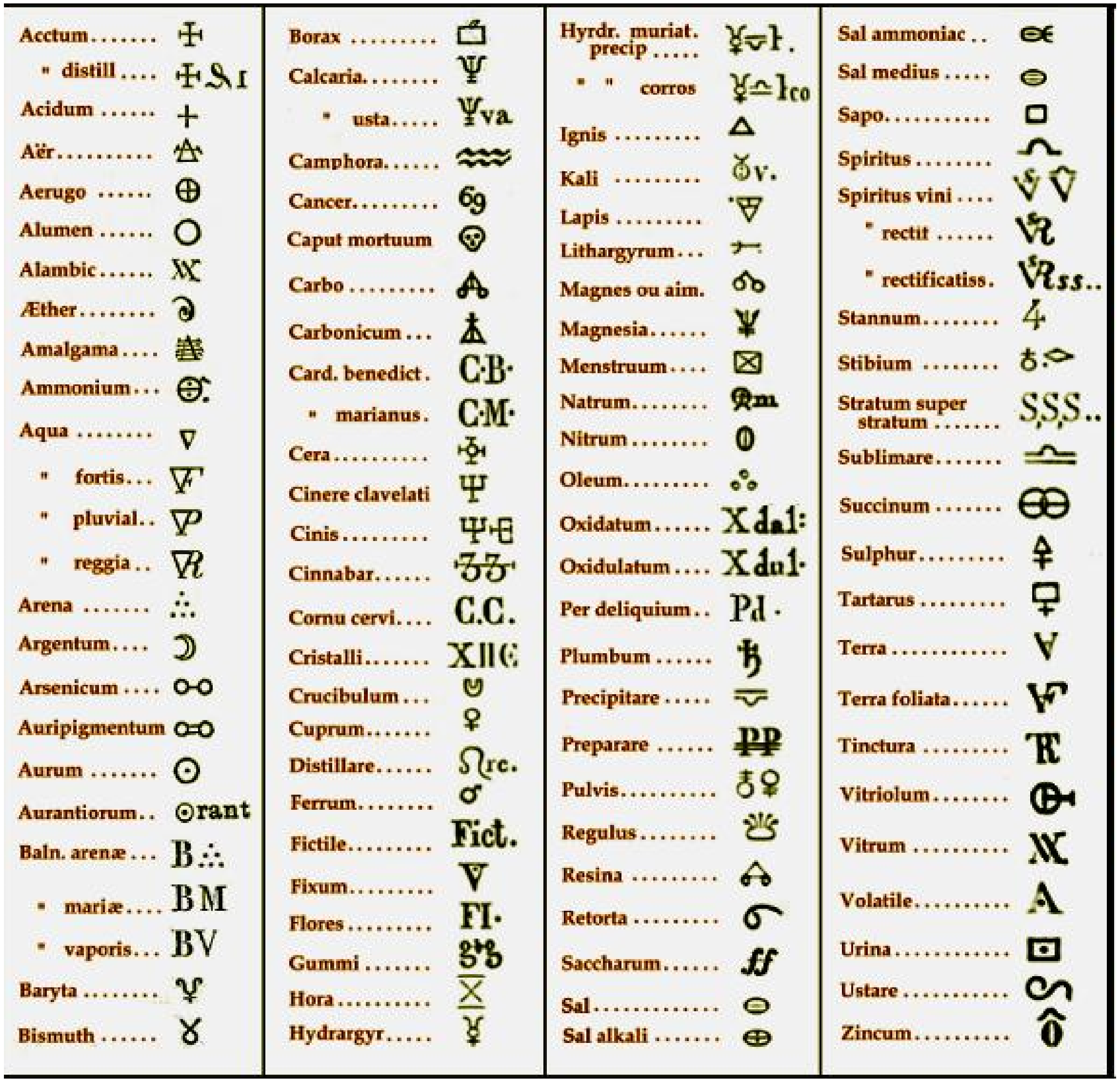
eg: CoSn / Sn

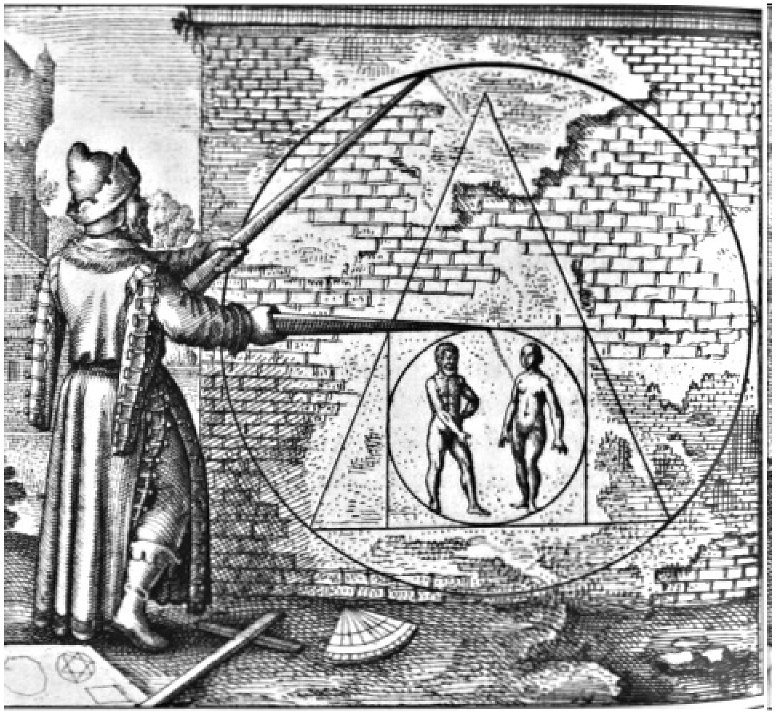
- self flux
- $Co_{0.20}Sn_{0.80}$
- 1050 °C → 650 °C

Before we continue….When I taught this nine years ago, I was accused of

engaging in

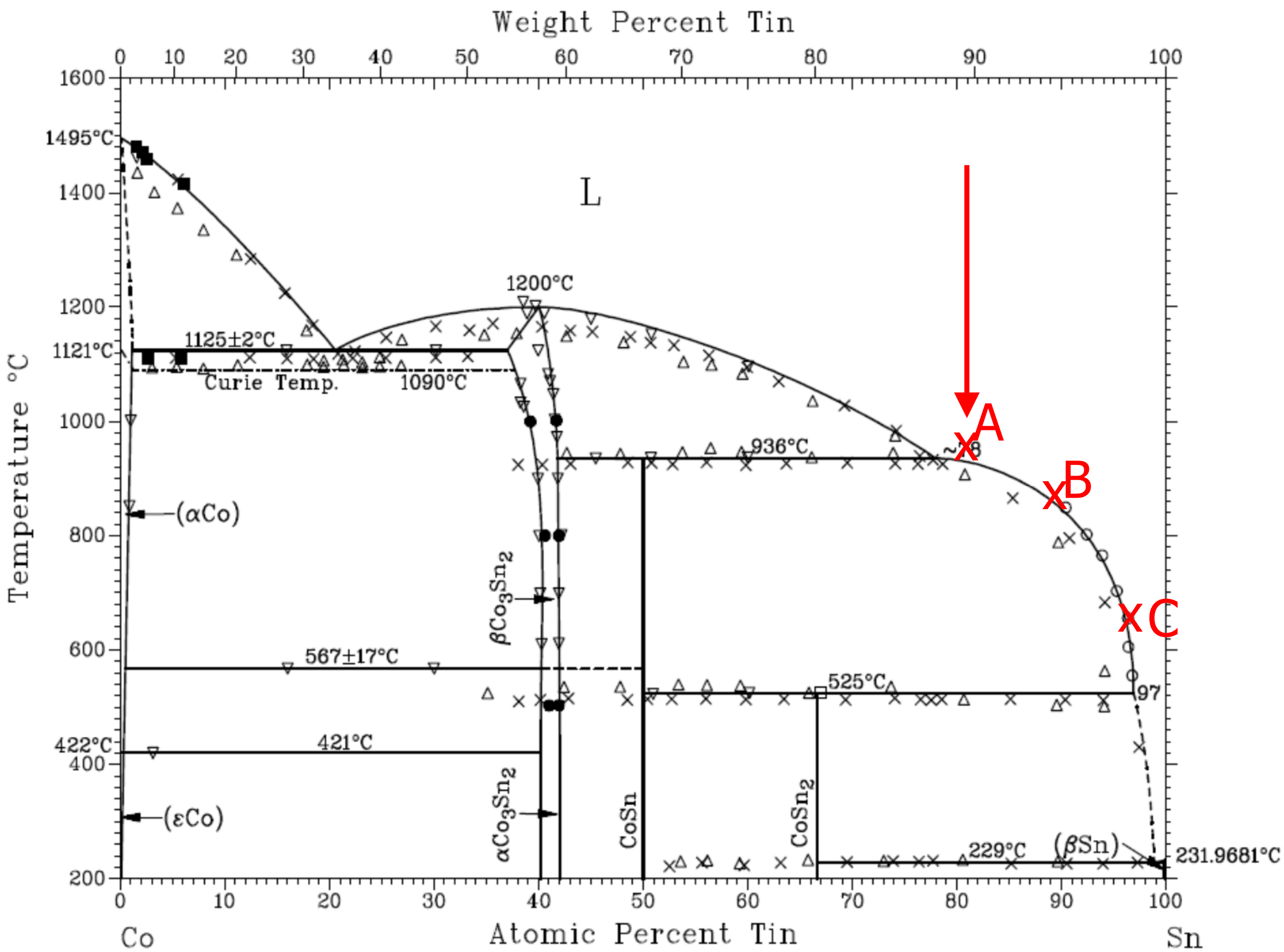
Before we continue….When I taught this nine years ago, I was accused of engaging in *ALCHEMY!!!!*





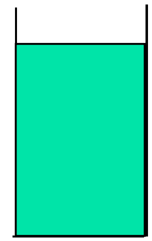
This is associated with the apparent change in the stoichiometry of the liquid as we cool into a two phase region.

Even though we start with a $Co_{20}Sn_{80}$ solution at high temperature, we end us with a nearly pure Sn solution at low temperature.



How does this work? We can not change Co into Sn!!!

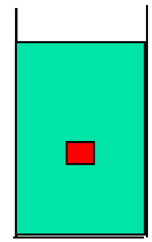
As we grow CoSn phase the remaining liquid becomes more Sn rich.



A

Liquid: 80% Sn

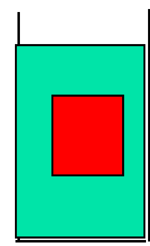
Solid: None



B

Liquid: 87% Sn

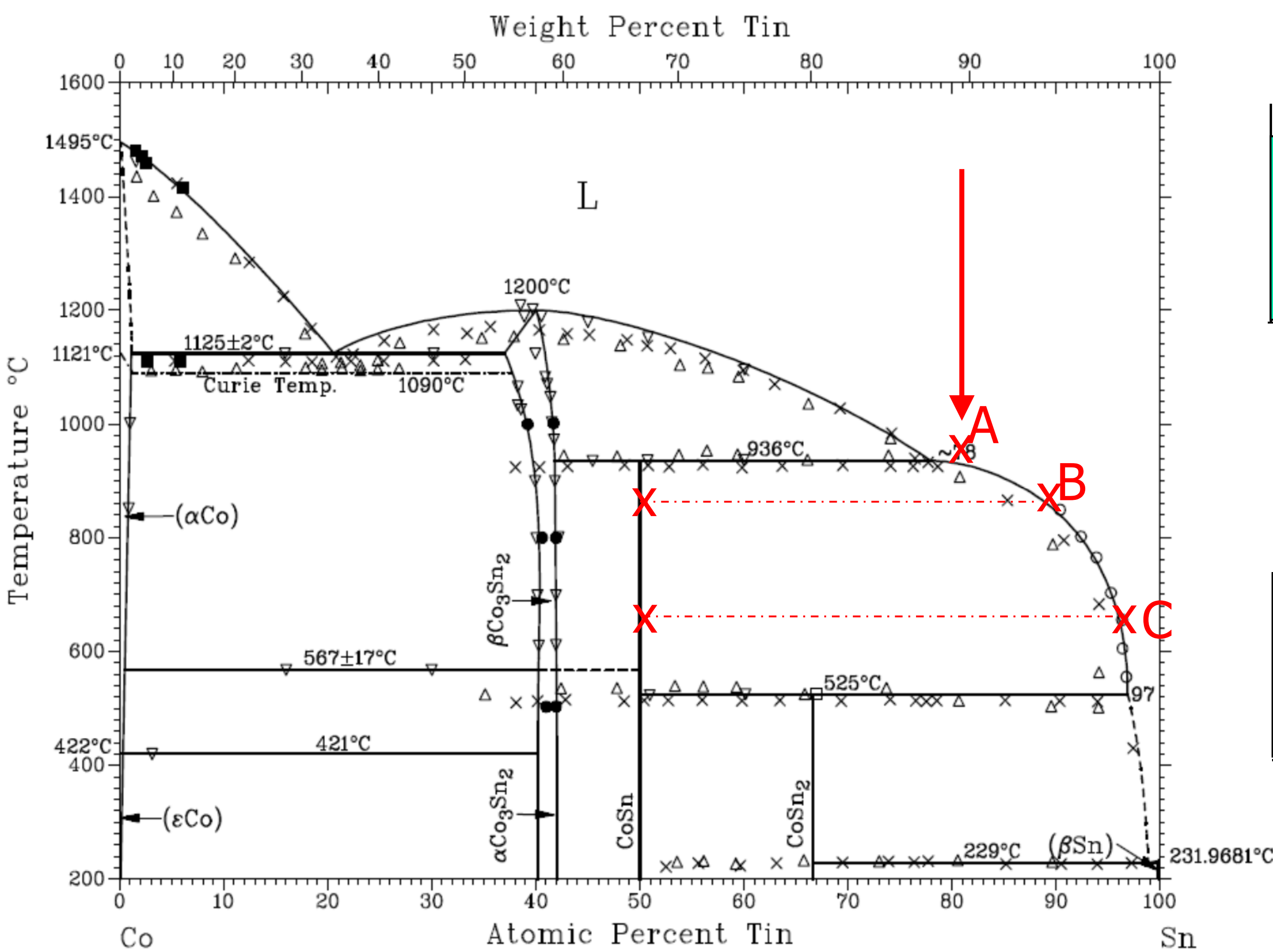
Solid: CoSn



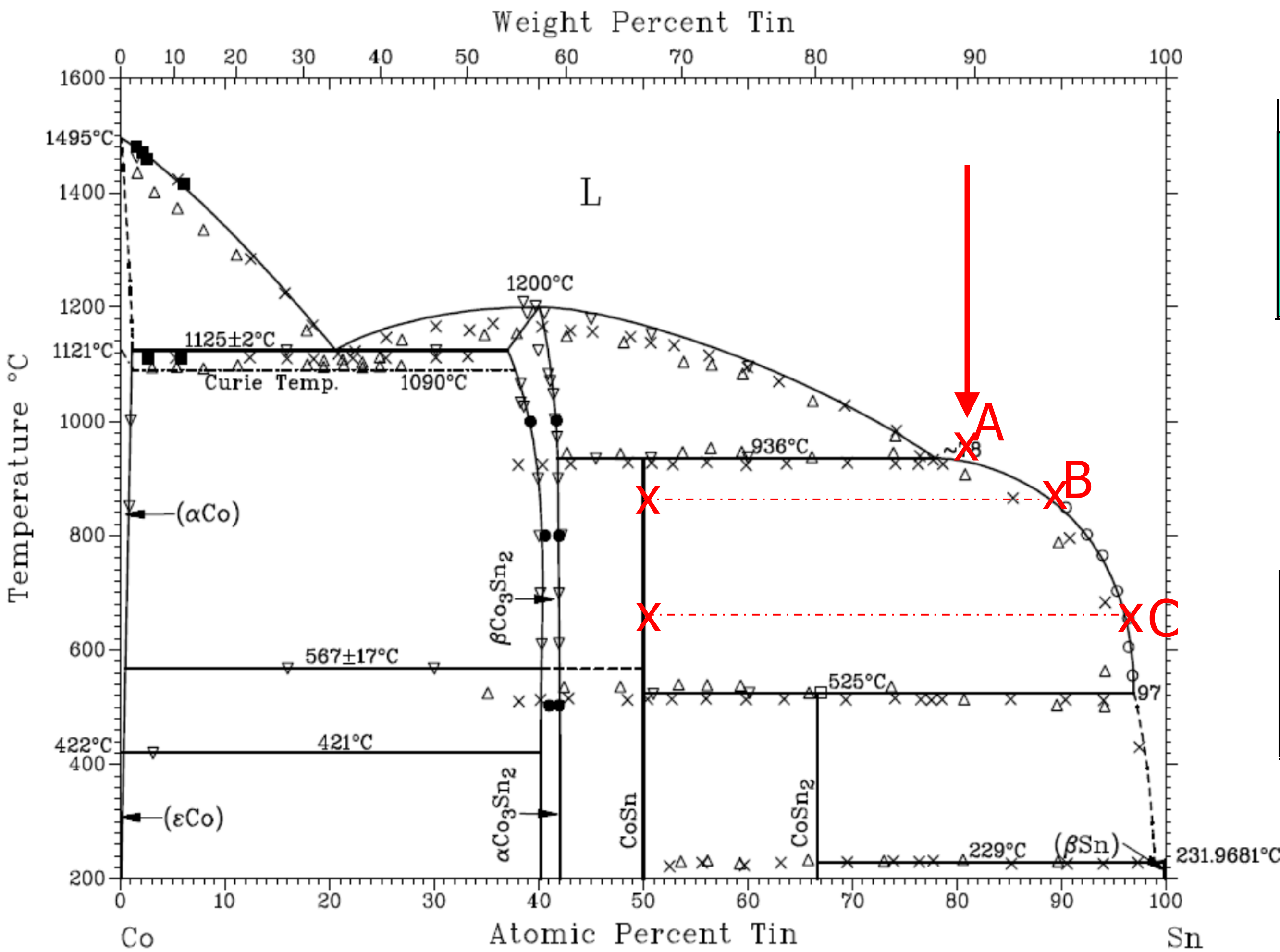
C

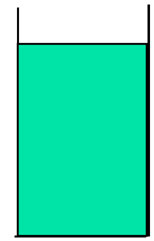
Liquid: 95% Sn

Solid: CoSn



The composition of liquid changes BECAUSE we are crystallizing a phase different from initial melt stoichiometry

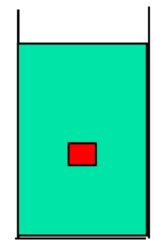




A

Liquid: 80% Sn

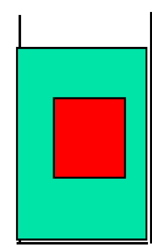
Solid: None



B

Liquid: 87% Sn

Solid: CoSn



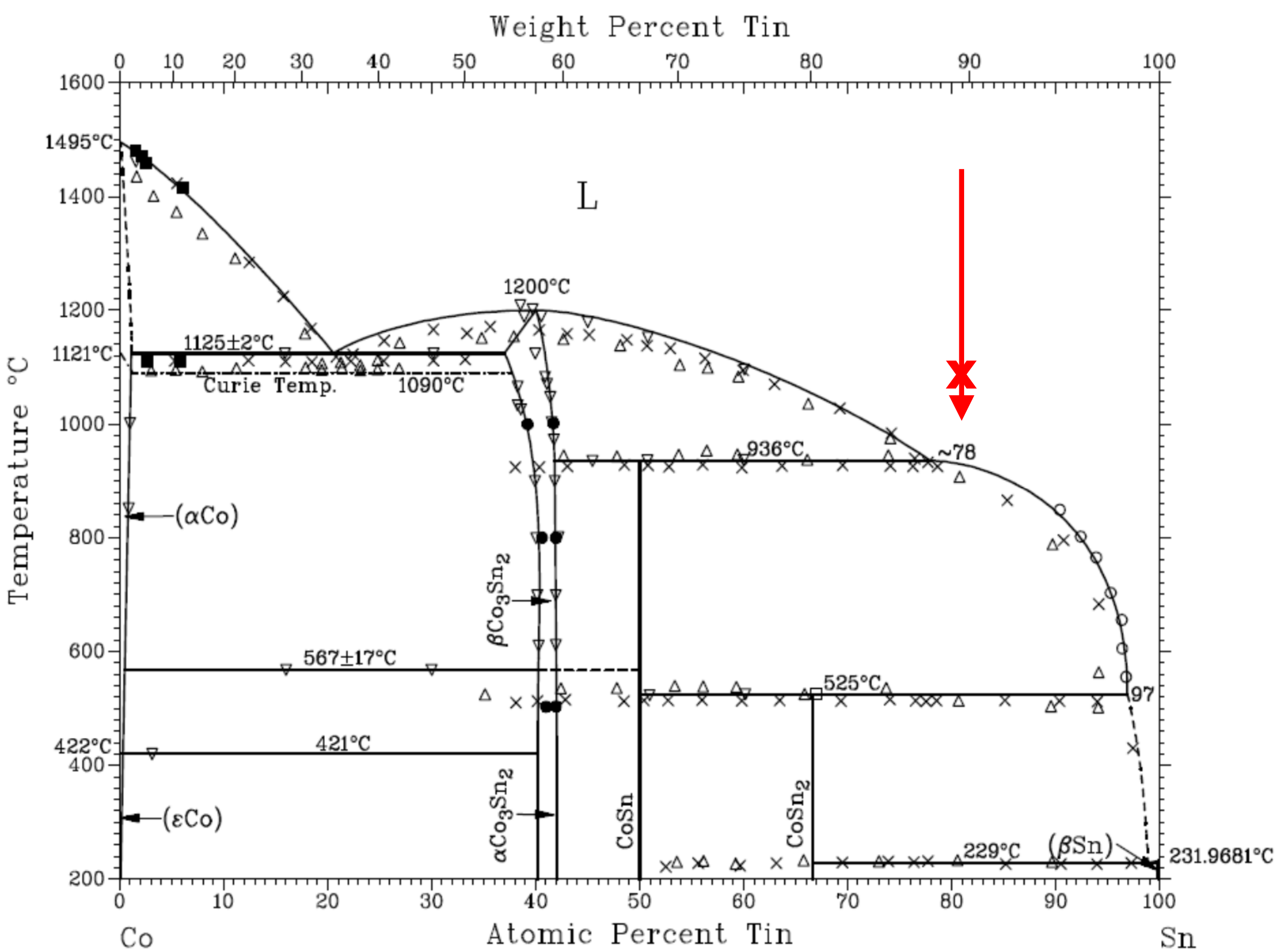
C

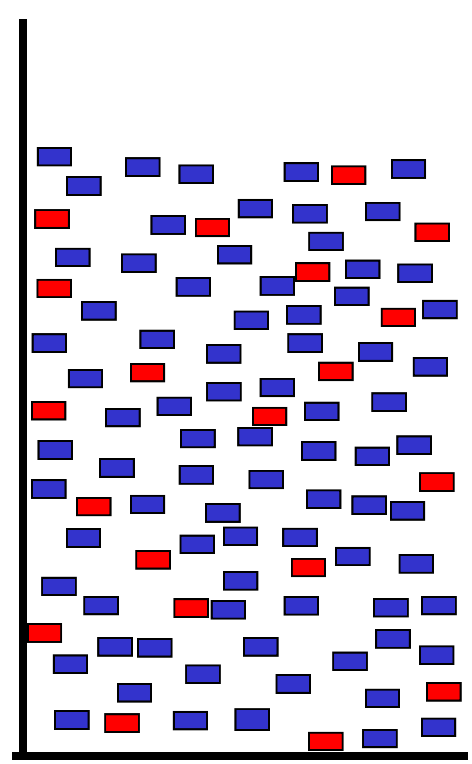
Liquid: 95% Sn

Solid: CoSn

We can also think about this in terms of atoms….

Let's have Sn blue and Co red in the mix to right. There are 80 Sn and 20 red blocks.

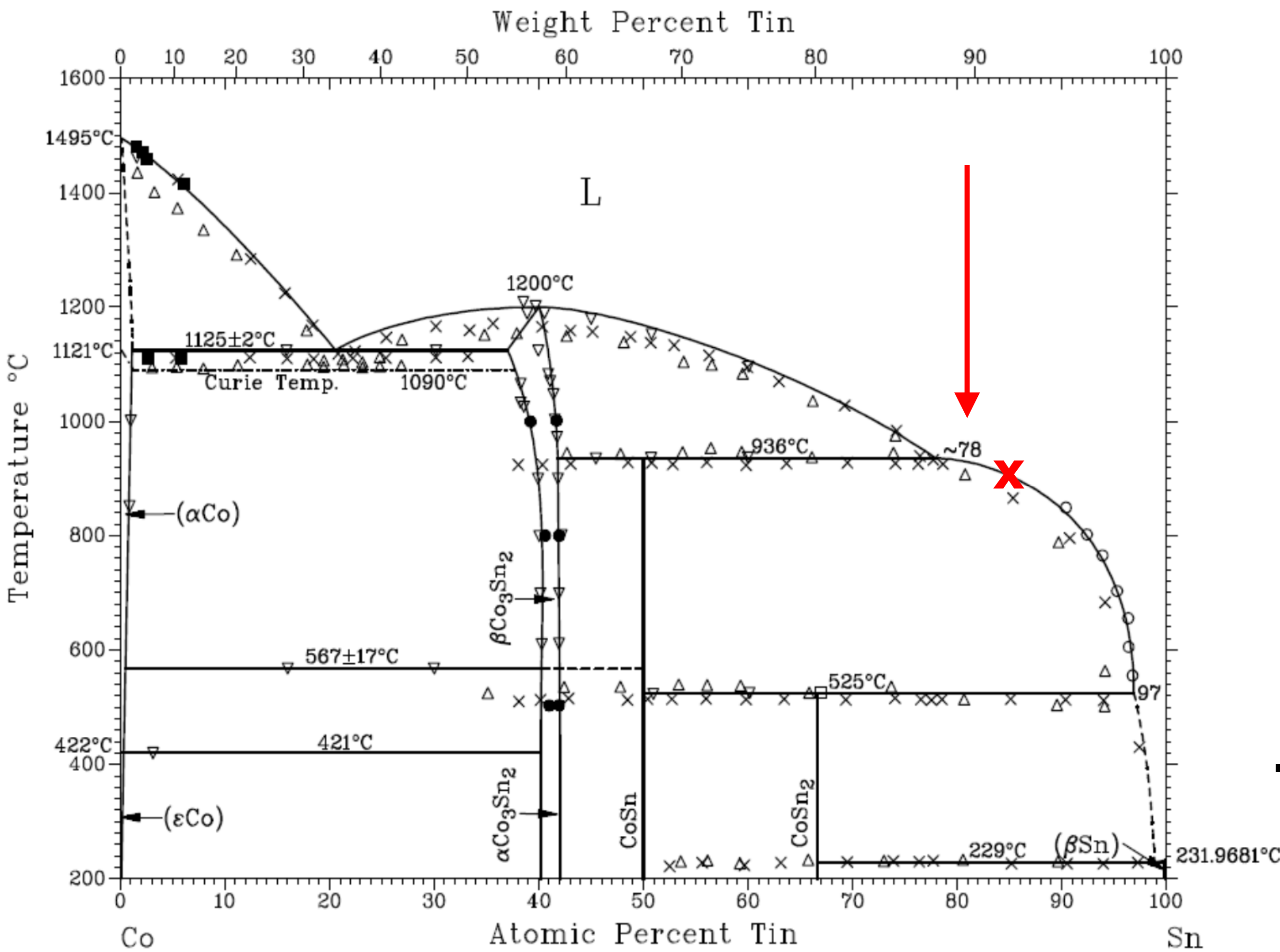


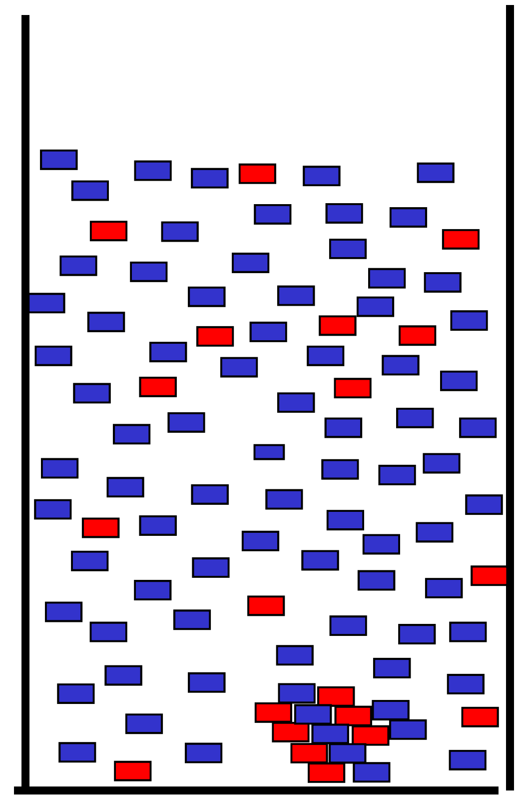


In single phase, liquid state

We can also think about this in terms of atoms….

Let's have Sn blue and Co red in the mix to right.
There are 80 Sn and 20 red blocks.

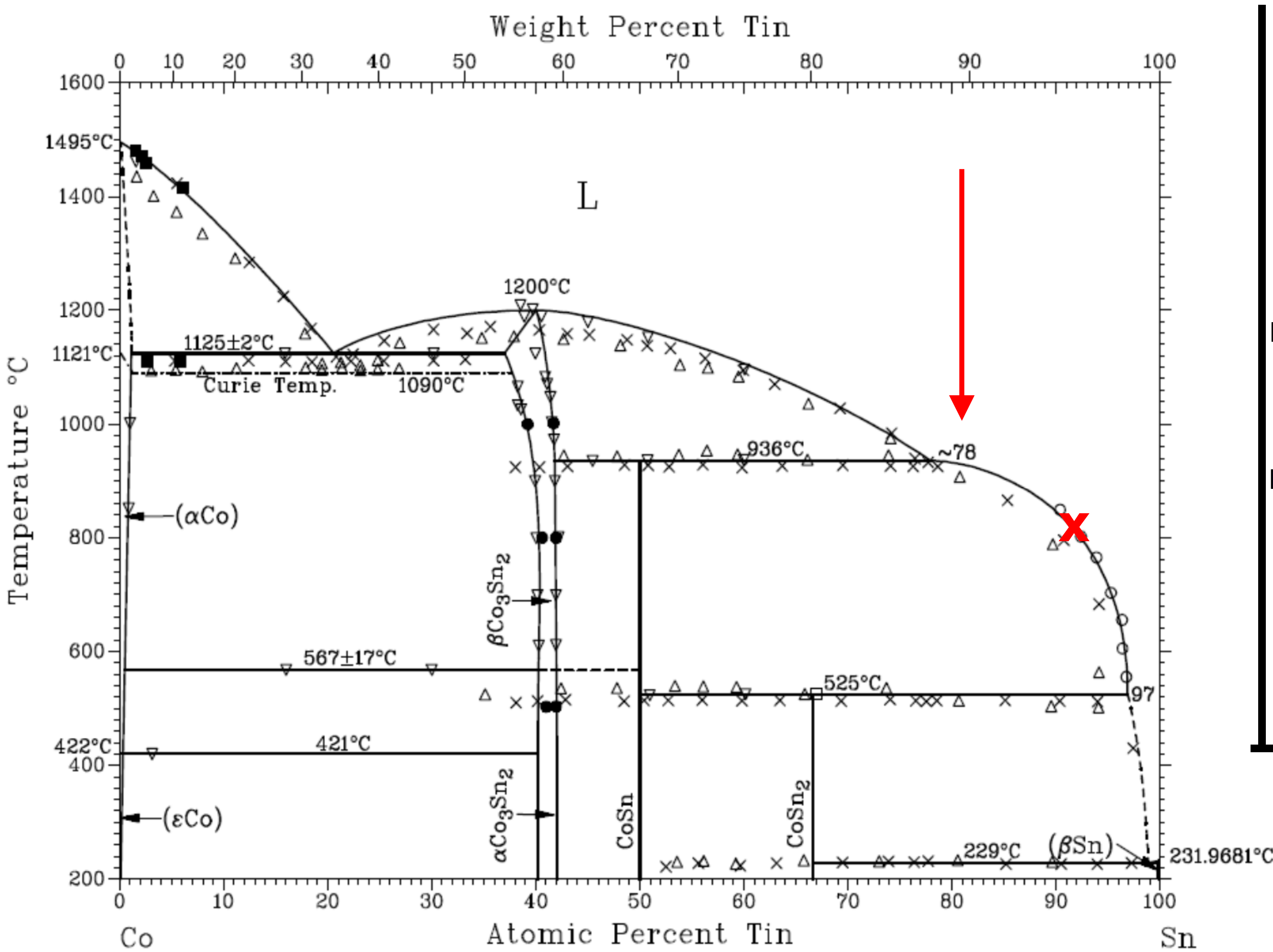


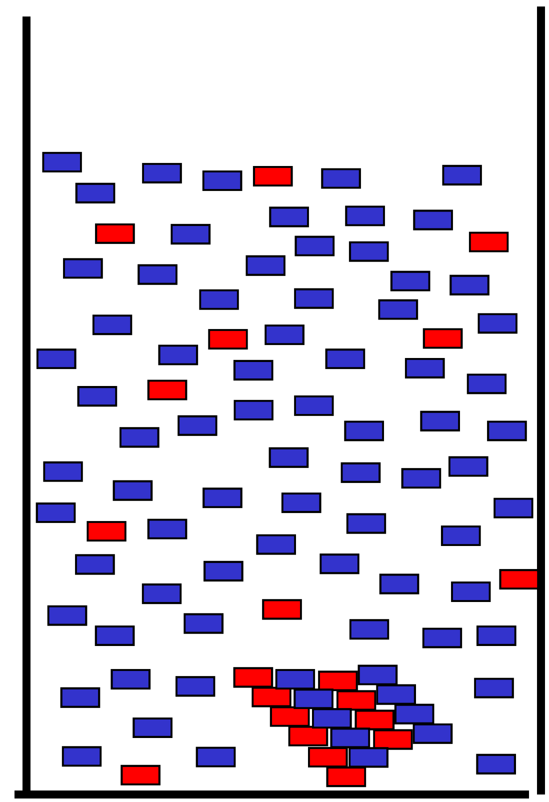


Just starting to grow CoSn

We can also think about this in terms of atoms….

Let's have Sn blue and Co red in the mix to right.
There are 80 Sn and 20 red blocks.

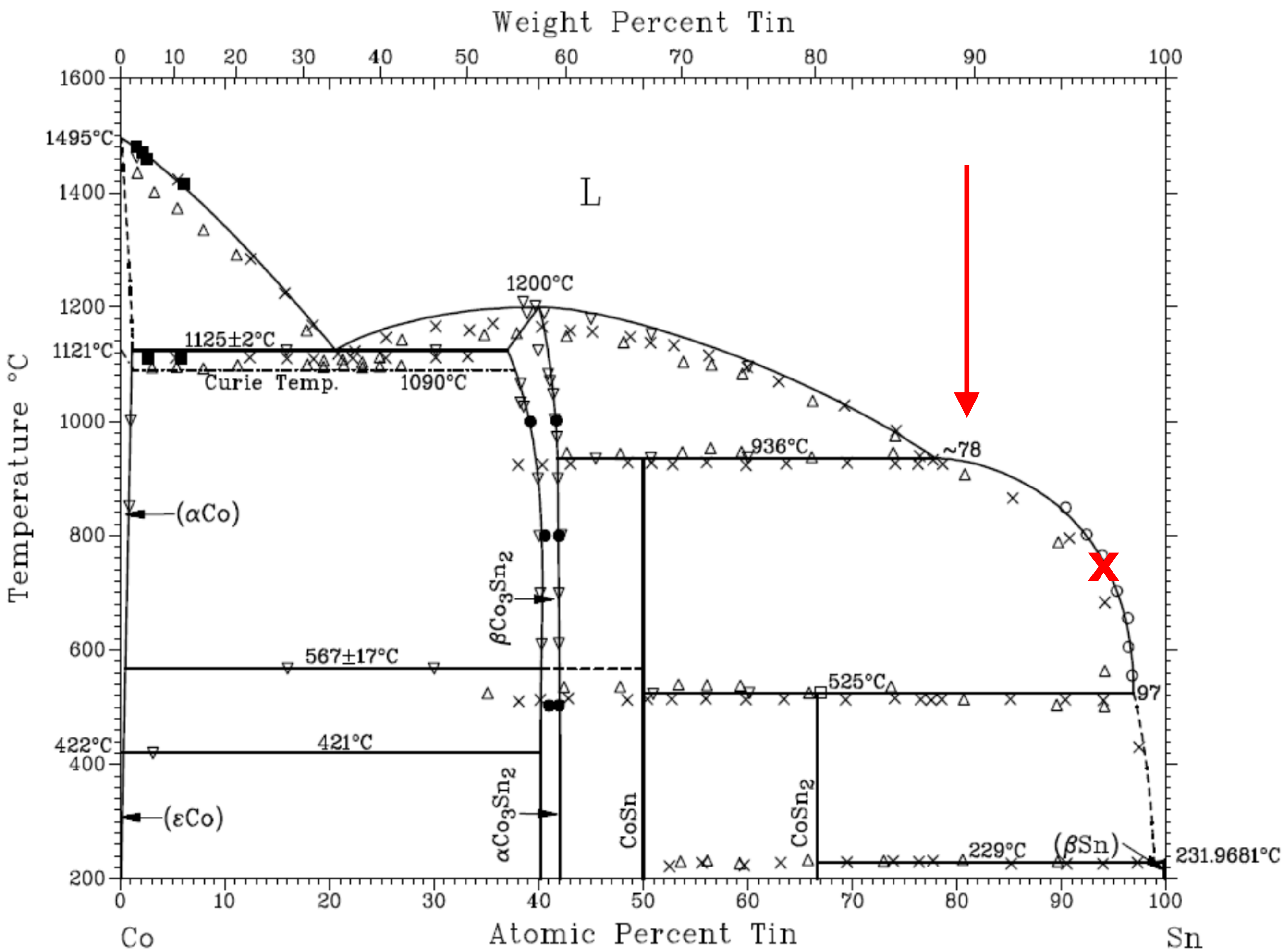


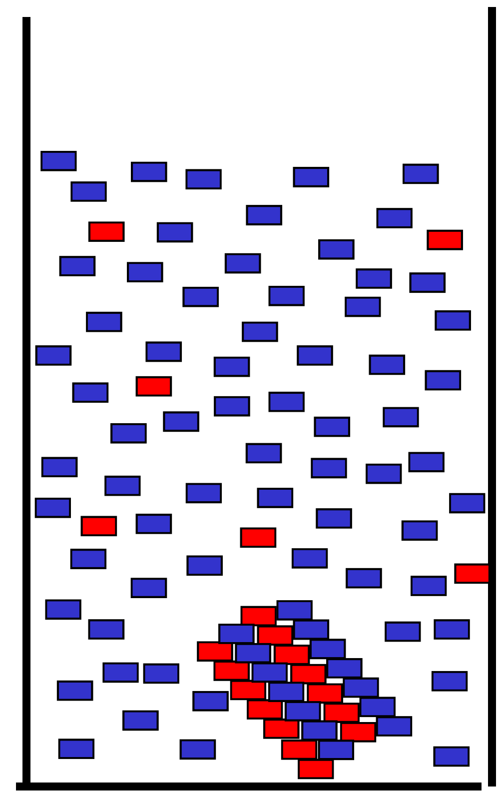


Note: as CoSn grows less Co in liquid

We can also think about this in terms of atoms….

Let's have Sn blue and Co red in the mix to right.
There are 80 Sn and 20 red blocks.

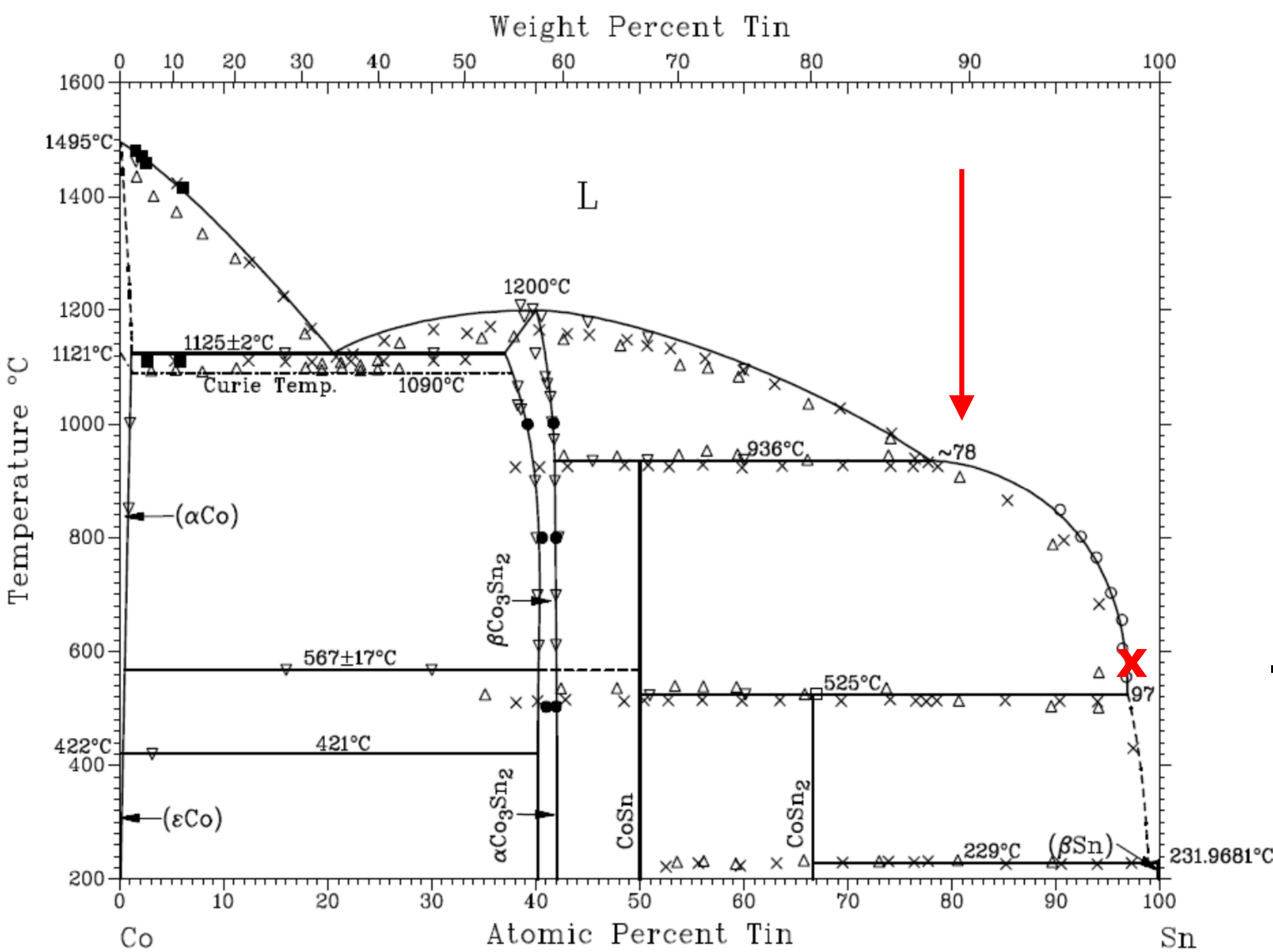


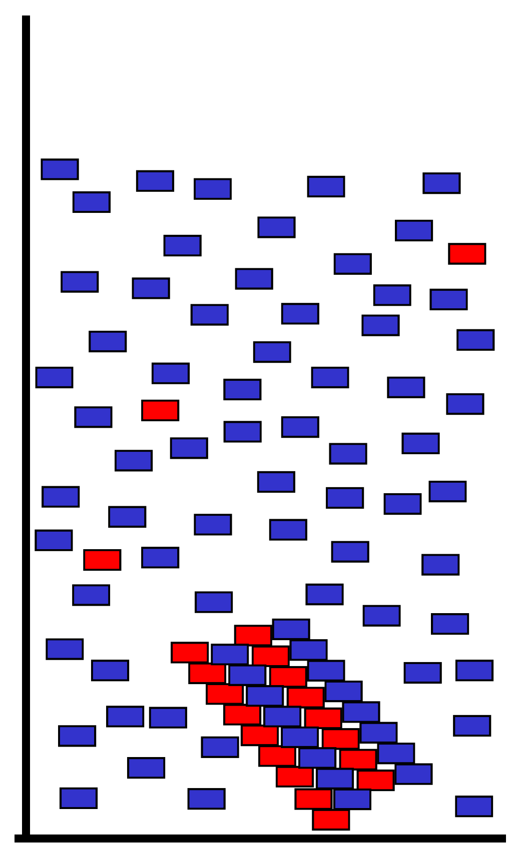


x on liquidus line gives composition of remaining liquid

We can also think about this in terms of atoms….

Let’s have Sn blue and Co red in the mix to right.
There are 80 Sn and 20 red blocks.

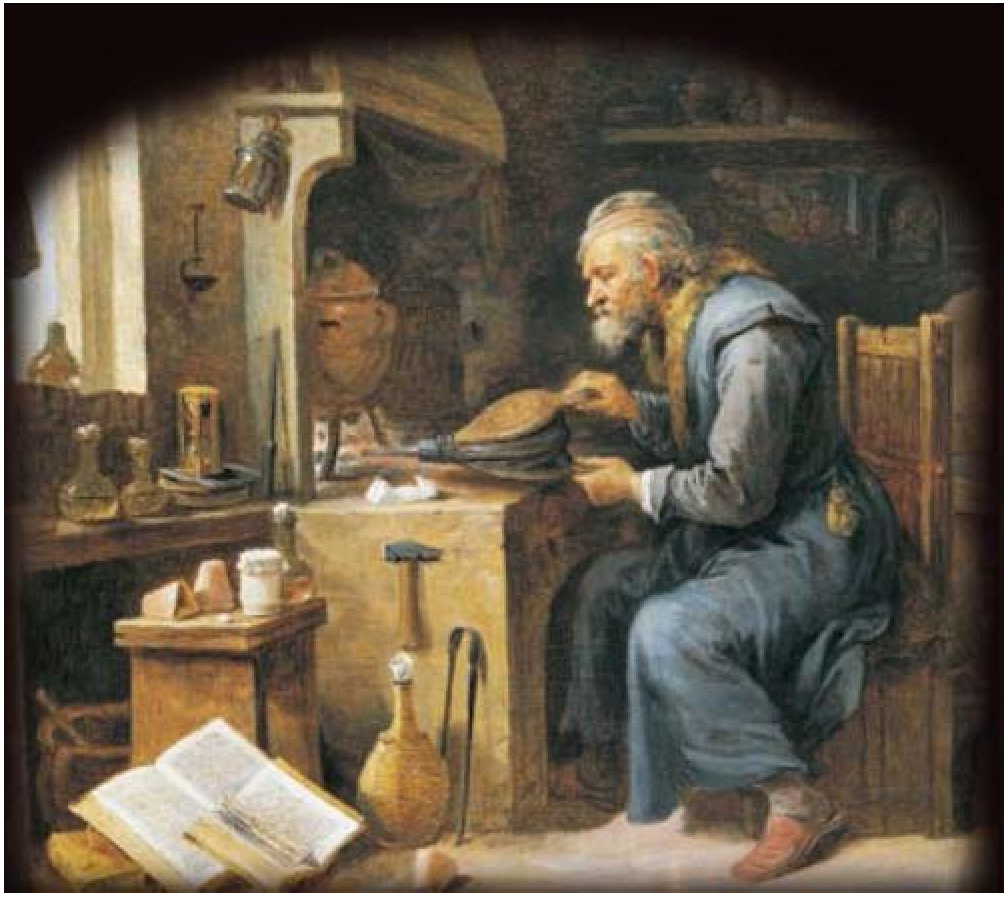


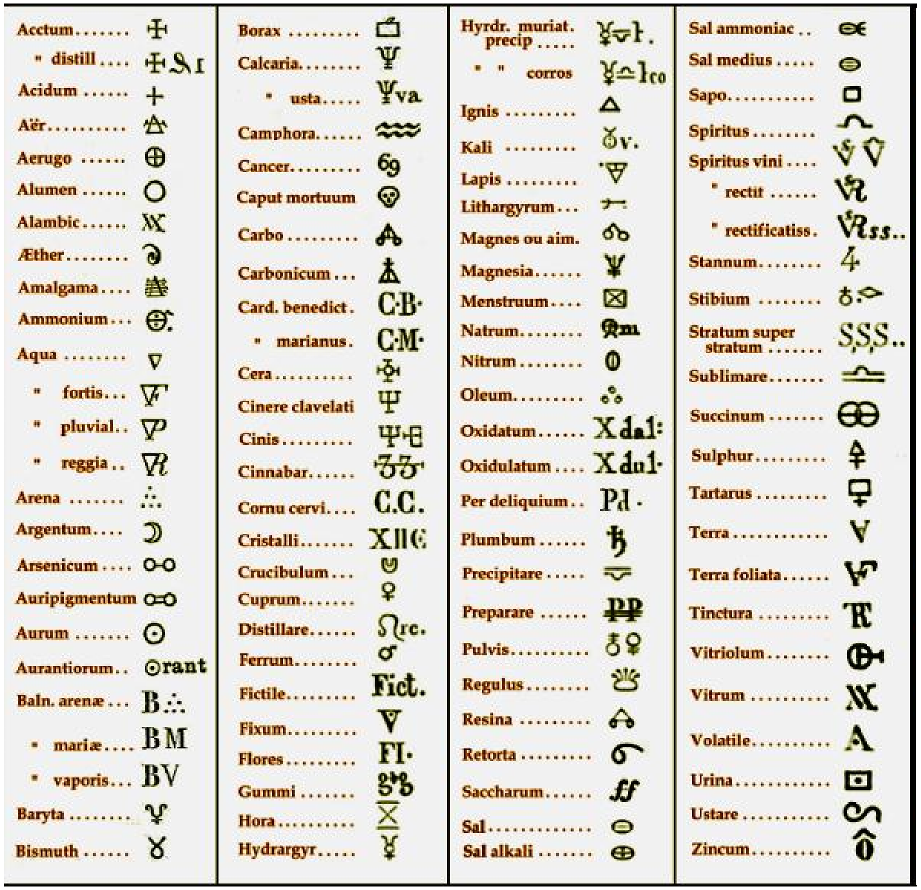


CoSn crystal has consumed most of initial Co. Liquid is almost pure Sn

Not *ALCHEMY,* just old fashion chemistry.

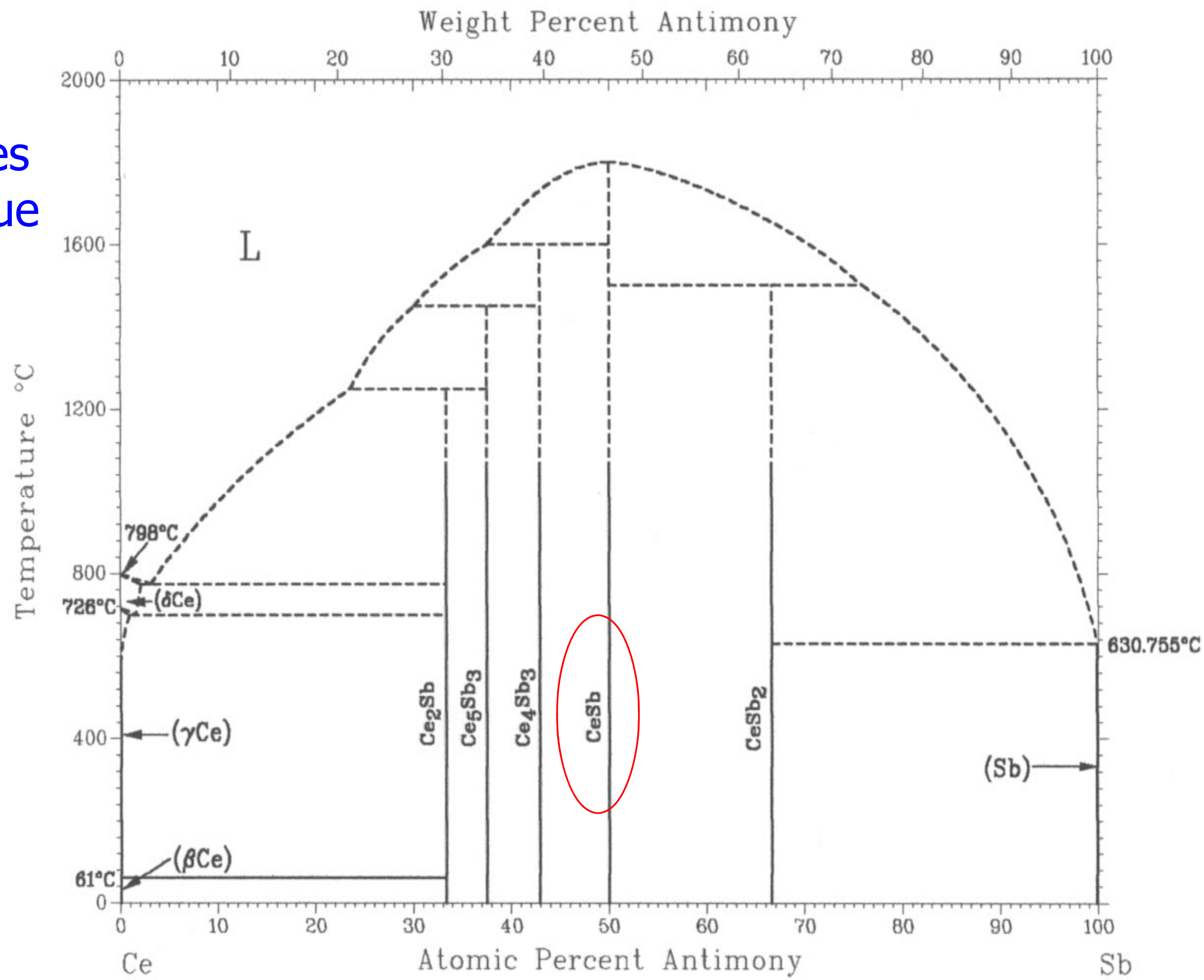
By the way, it is interesting to study history of chemistry as it emerged from alchemy into systematic science; very active debate and evolution of science. Look at names of elements to get some of this flavor.





OK, now higher melts: what about growth of the congruently melting compound, CeSb?

This is tricky if done just out of the binary: very high temperatures and lots of defects (due to vapor pressure and entropy).

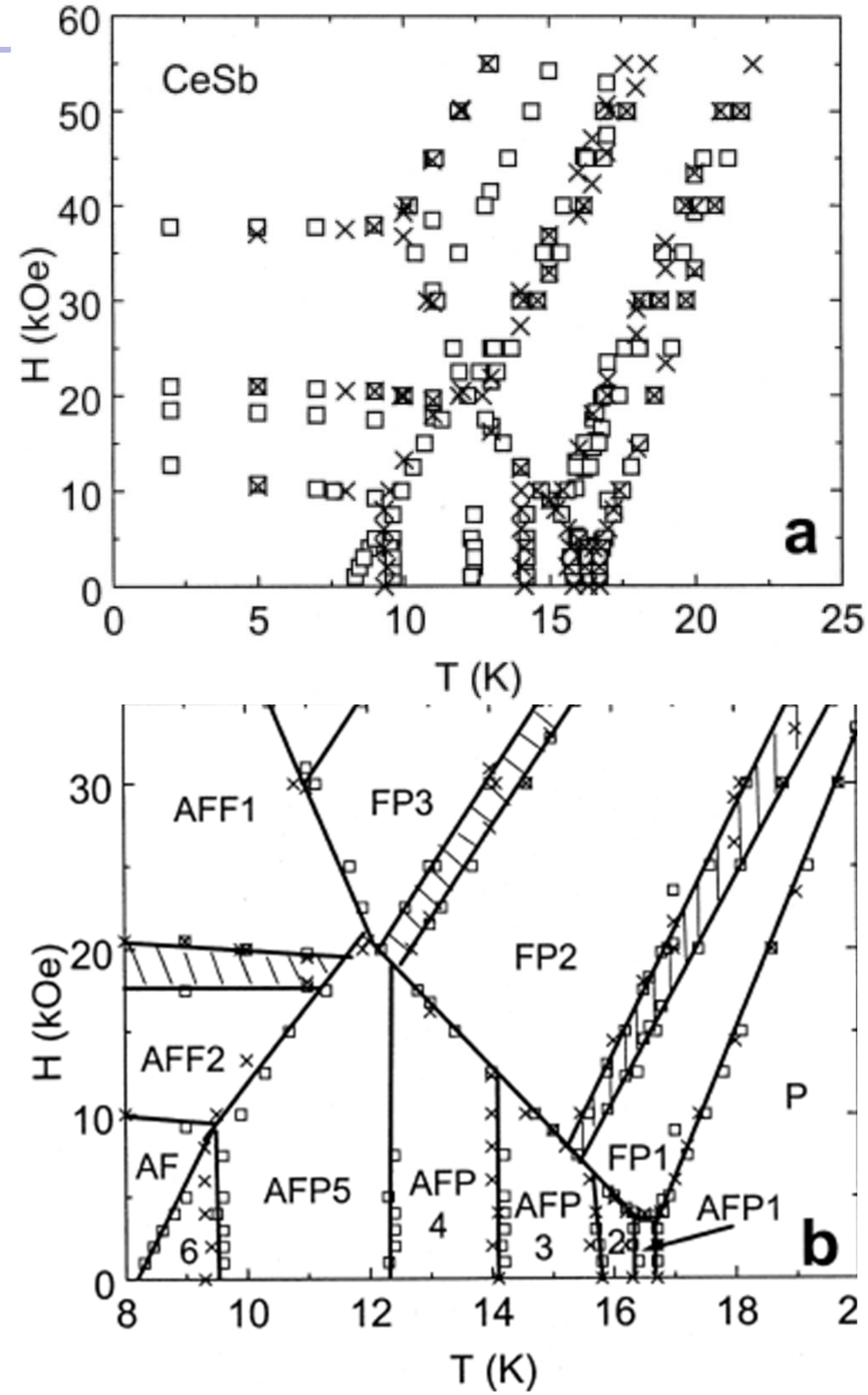


But why would you want to grow this compound anyway?

Multiple transitions in temperature and field

CeSb

M(H), M(T), ρ(H) and ρ(T) data can be used to assemble an H-T phase diagram of fantastic detail. This system was studied extensively in the 70's and 80's by several neutron scattering groups as well as serving as the inspiration for the ANNNI model.



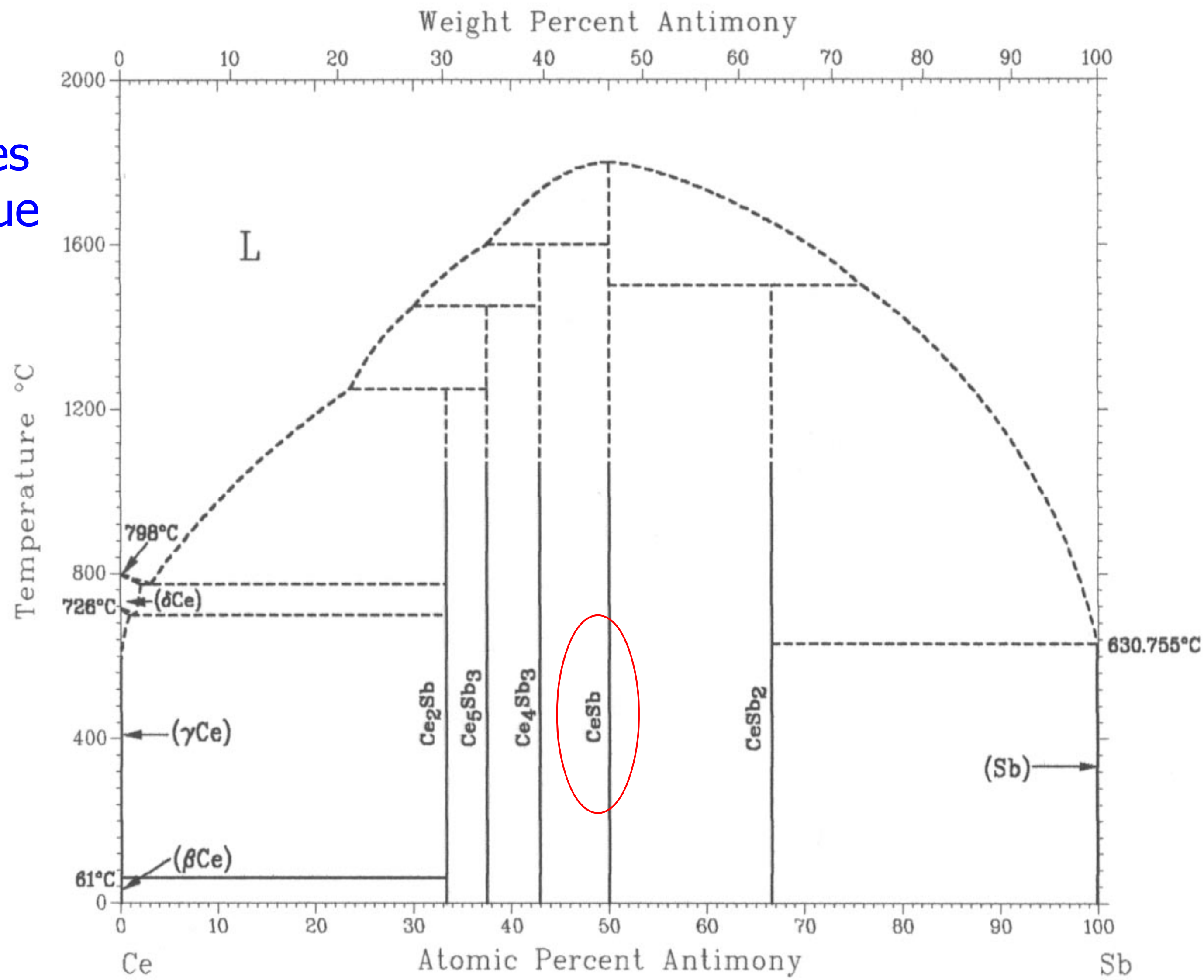
T.A. Wiener, P.C. Canfield / Journal of Alloys and Compounds 303–304 (2000) 505–508

OK, now higher melts: what about growth of the congruently melting compound, CeSb?

This is tricky if done just out of the binary: very high temperatures and lots of defects (due to vapor pressure and entropy).

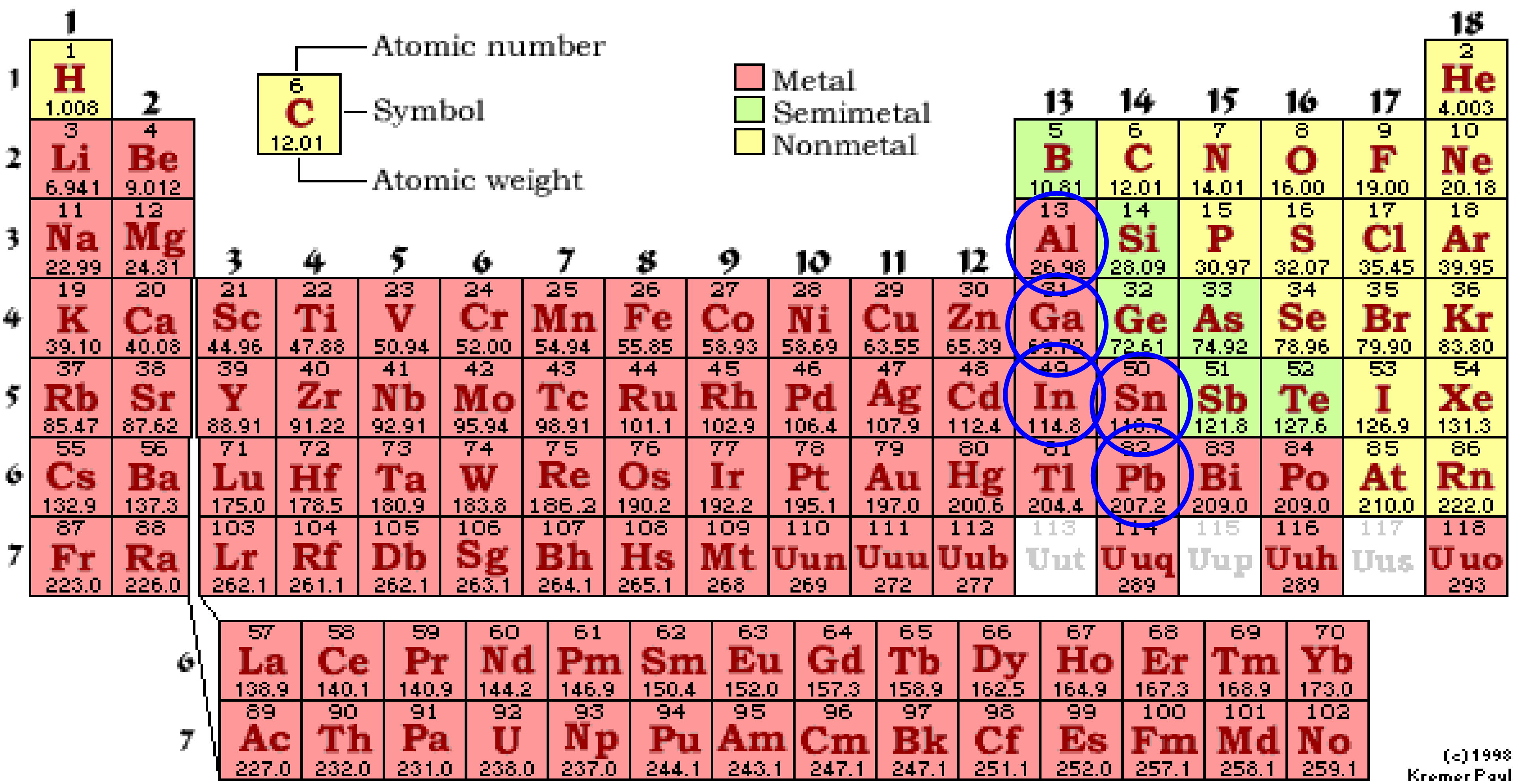
Can this be grown out of extra elements in manner similar to growing a salt out of water?

This question is the essence of flux growth.



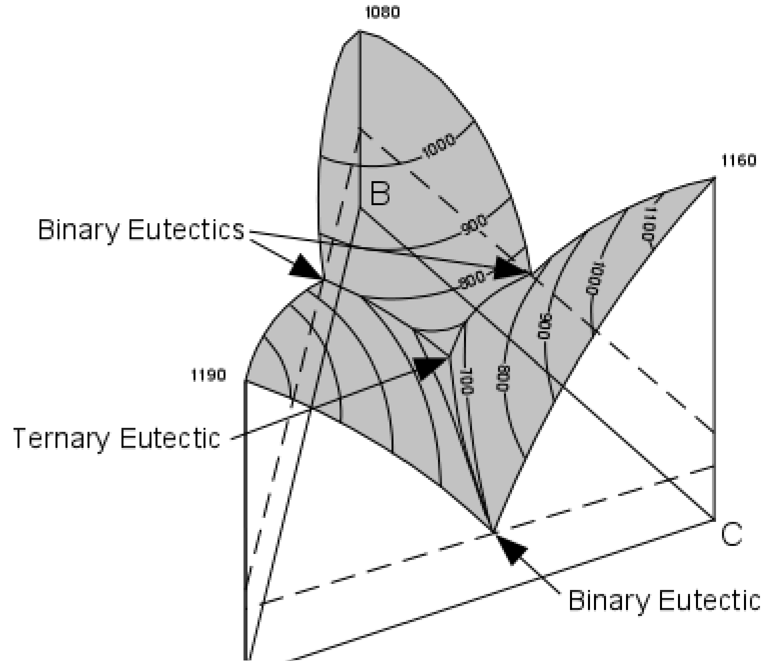
When I was first faced with this goal I simply tried several of the “usual suspects”, i.e. low melting elements that offered good solubility for both Ce and Sb.

Sn worked best



We are now talking about a ternary growth.

We have added another dimension.



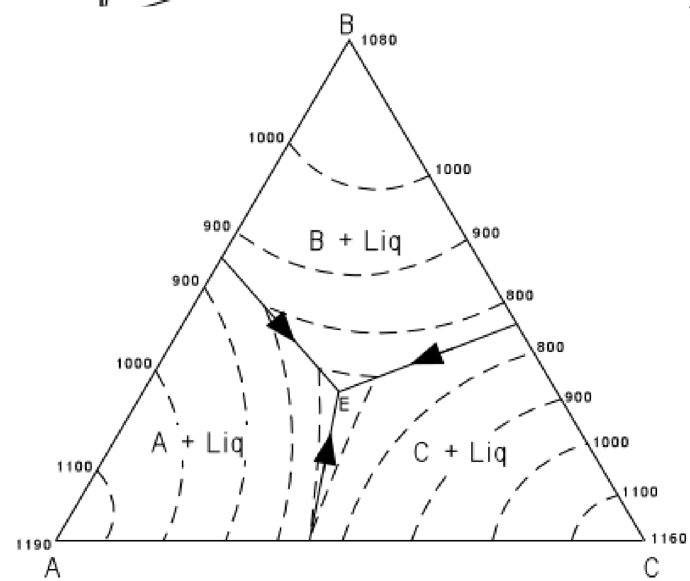


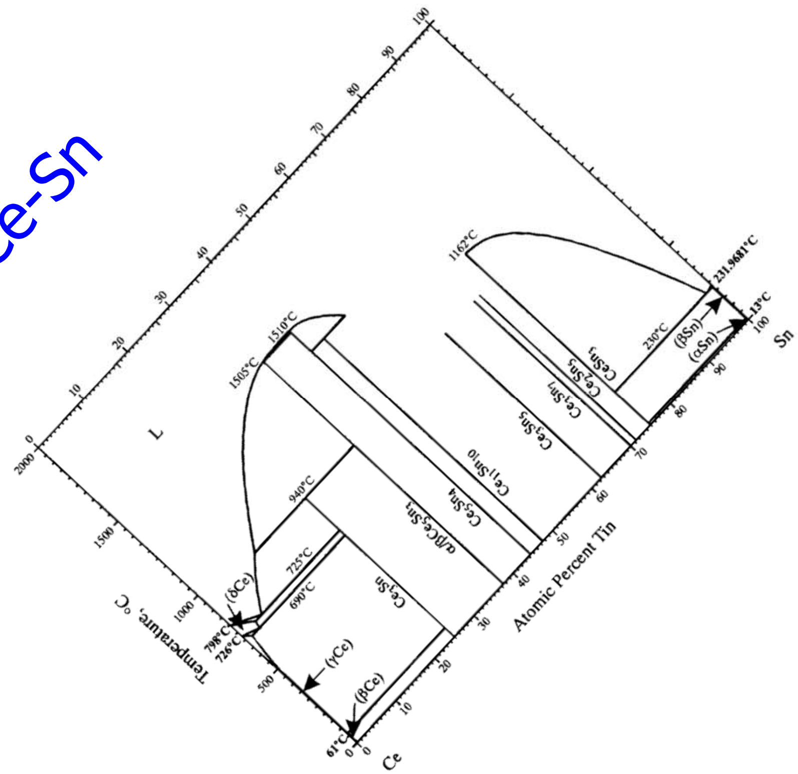
Figure 2

http://www.tulane.edu/~sanelson/geol212/ternaryphdiag.htm

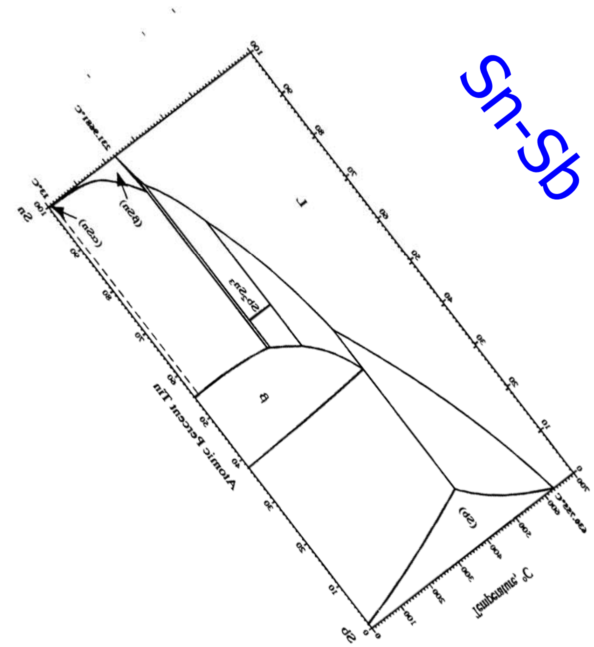
In many cases we are guessing how to ski blind, on a slope we can only speculate about.

Note, when the ternary diagram is known (rarely) it looks a lot like a topo-map.

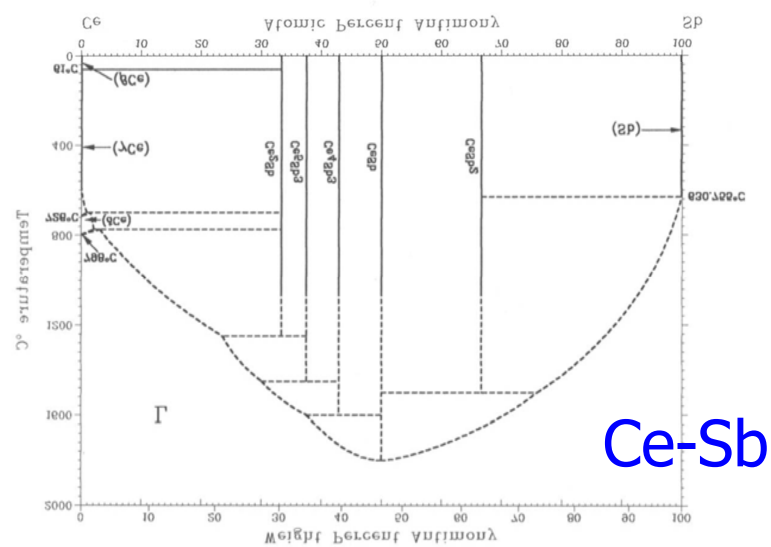
Ce-Sn



Sn-Sb

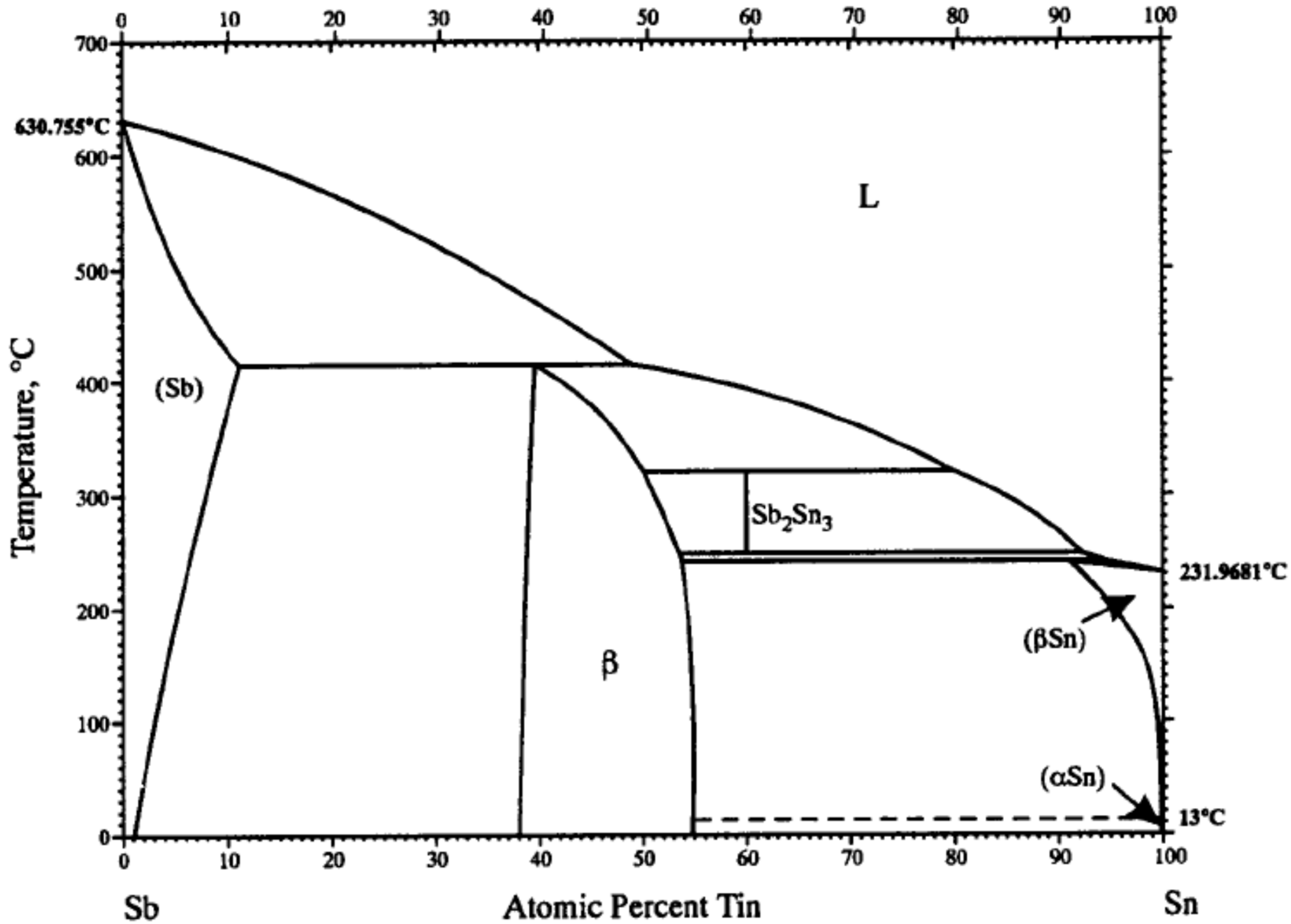


We can use binary phase diagrams to give sense of edges of the ternary.

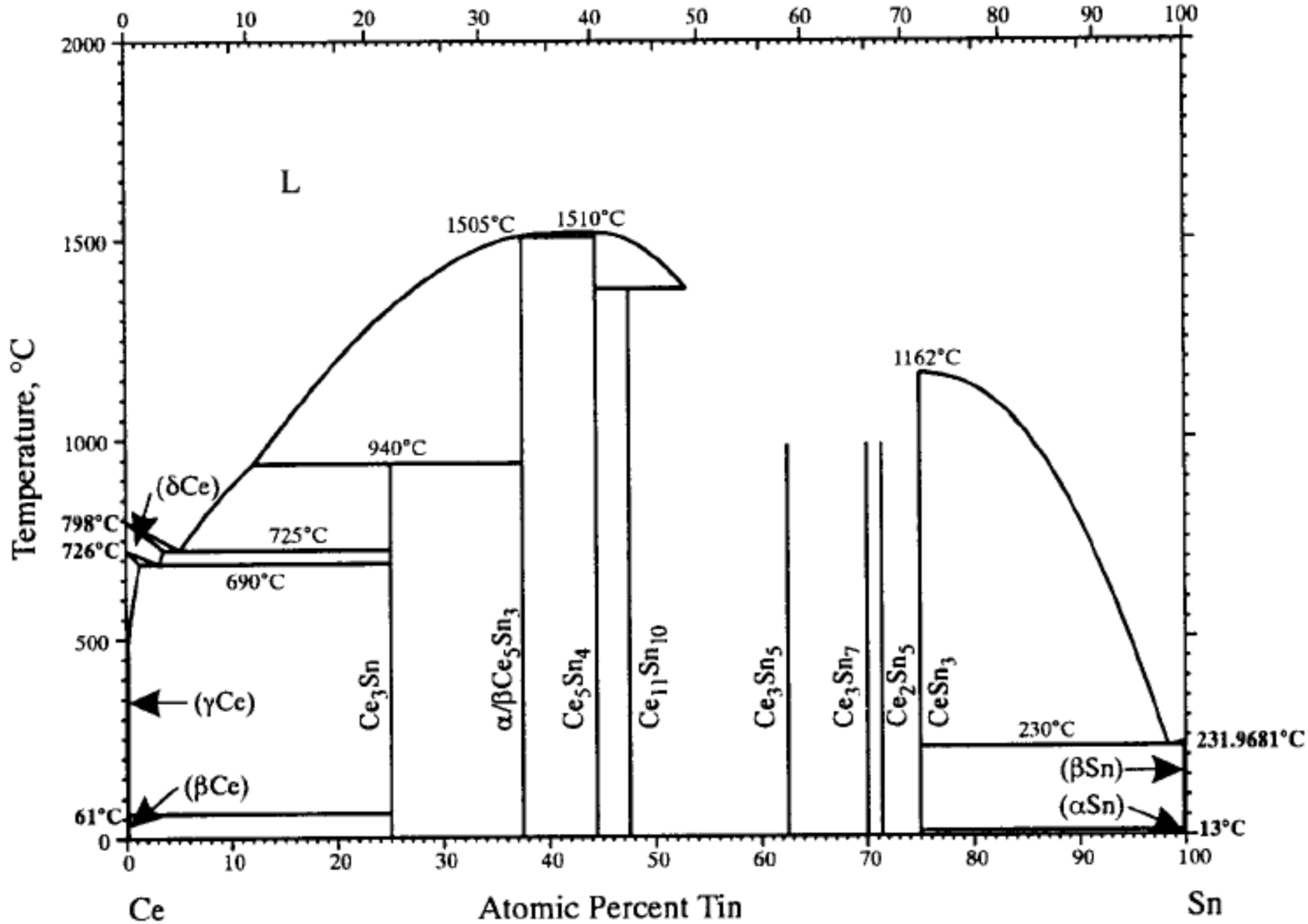


Ce-Sb

There are only low melting compounds of Sn and Sb



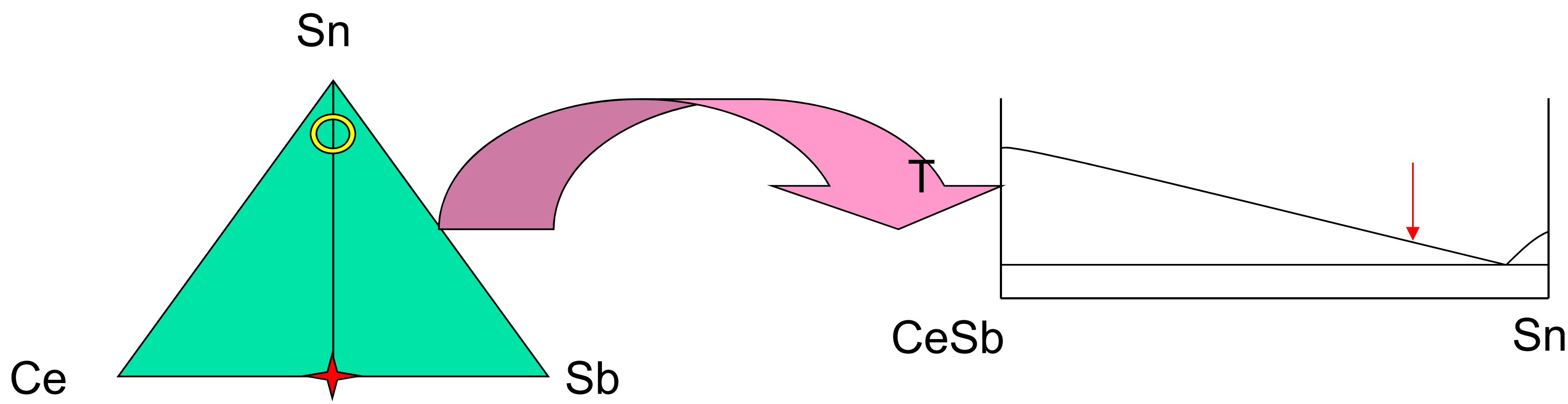
If the Ce is dilute in Sn, then we only worry about $CeSn_3$ at low temperatures.

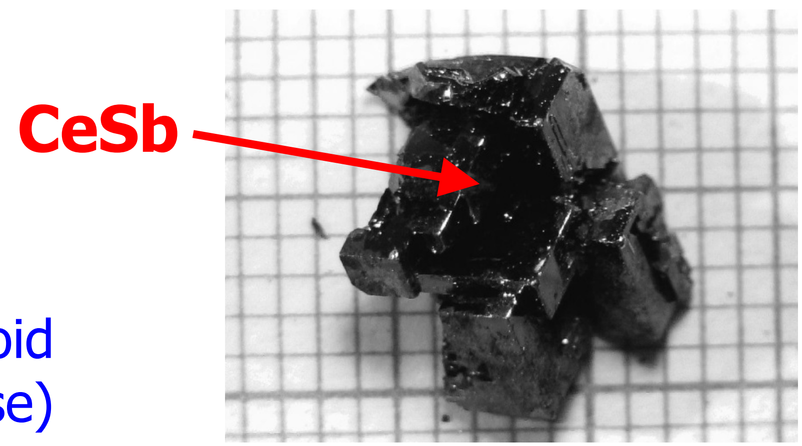


3^{rd} element flux

eg: CeSb / Sn

In this case we can think of this as a pseudo-binary cut through the Ce-Sb-Sn ternary phase diagram.

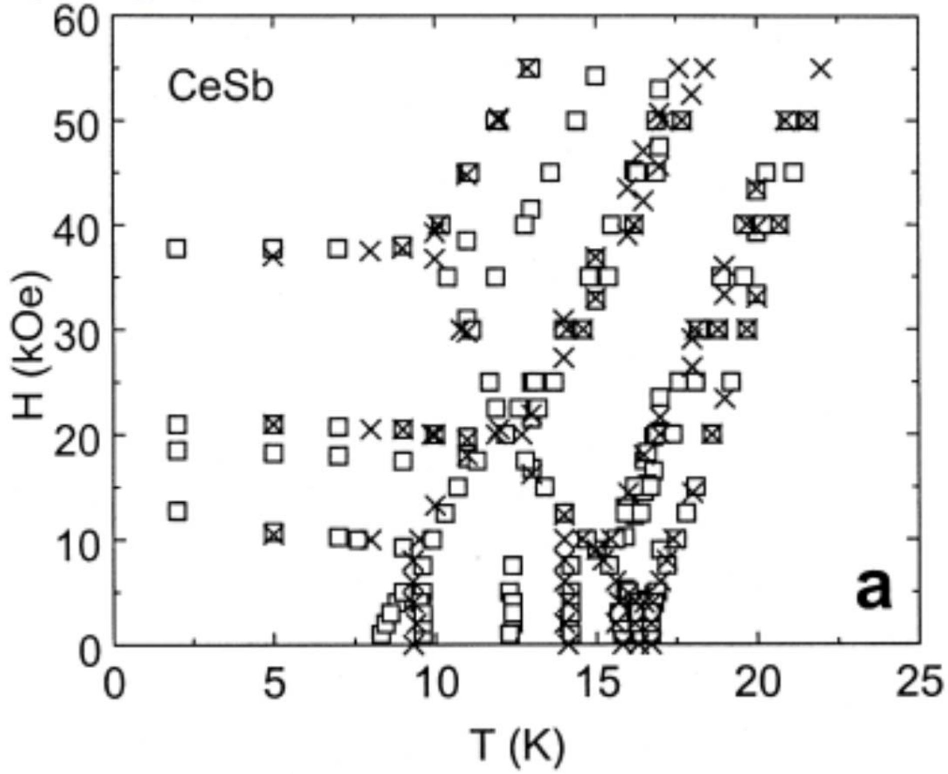




$(CeSb)_{0.05}Sn_{0.95}$

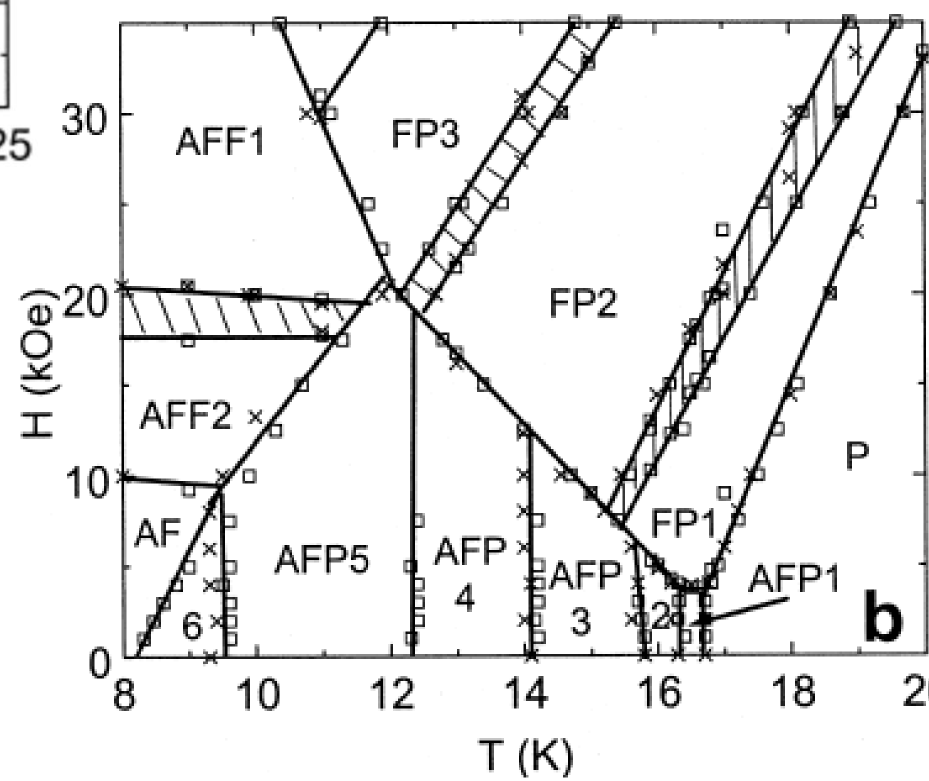
1150 °C → 800 °C

(Spin at 800 to avoid the $CeSn_3$ 2^{nd} phase)



Complex field and temperature dependent magnetism can be found in CeSb, especially very high purity single crystals grown out of Sn in this method.

M(H), M(T), ρ(H) and ρ(T) data can be used to assemble an H-T phase diagram of fantastic detail.



T.A. Wiener, P.C. Canfield / Journal of Alloys and Compounds 303–304 (2000) 505–508

For the rest of this lecture we will review other examples of growth design and implementation.   I will try to point out issues associated with:

Silica Softening---When using silica tubing you must respect T ~ 1200 C

Vapor pressure (attack)

Crucible stability

But before that do be aware of:

Toxicity of compounds before reaction

Toxicity of compounds after reaction

Check before you start

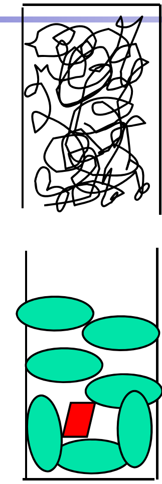
Expense of it all…Quartz, crucibles, elements….

Let's start with an example of a growth that appears to be easy, but requires attention to

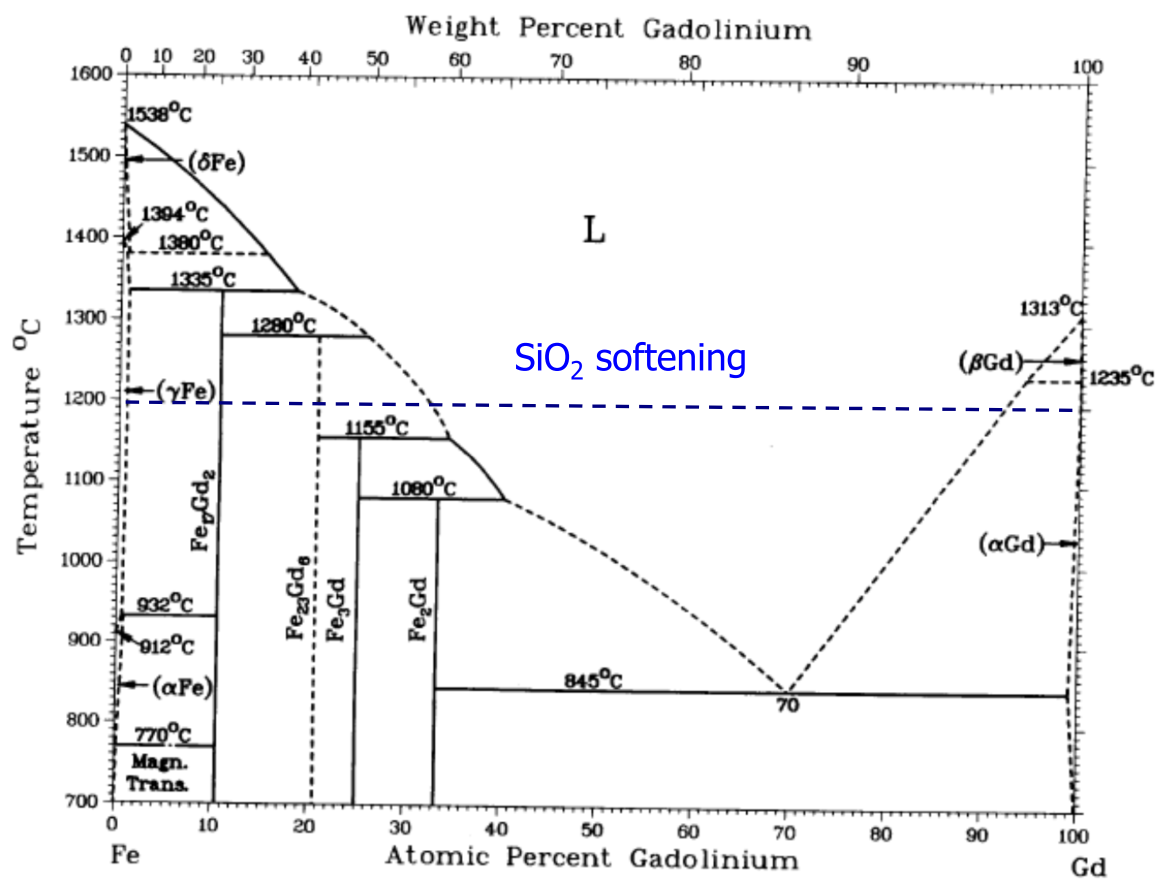
Silica Softening---When using silica tubing you must respect T ~ 1200 C

Crucible stability

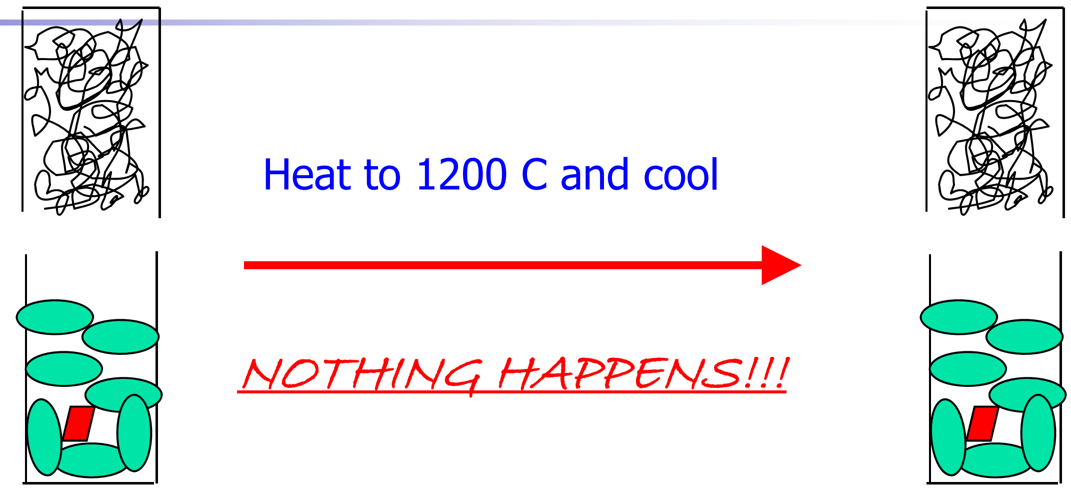
Let's try to grow $GdFe_2$.

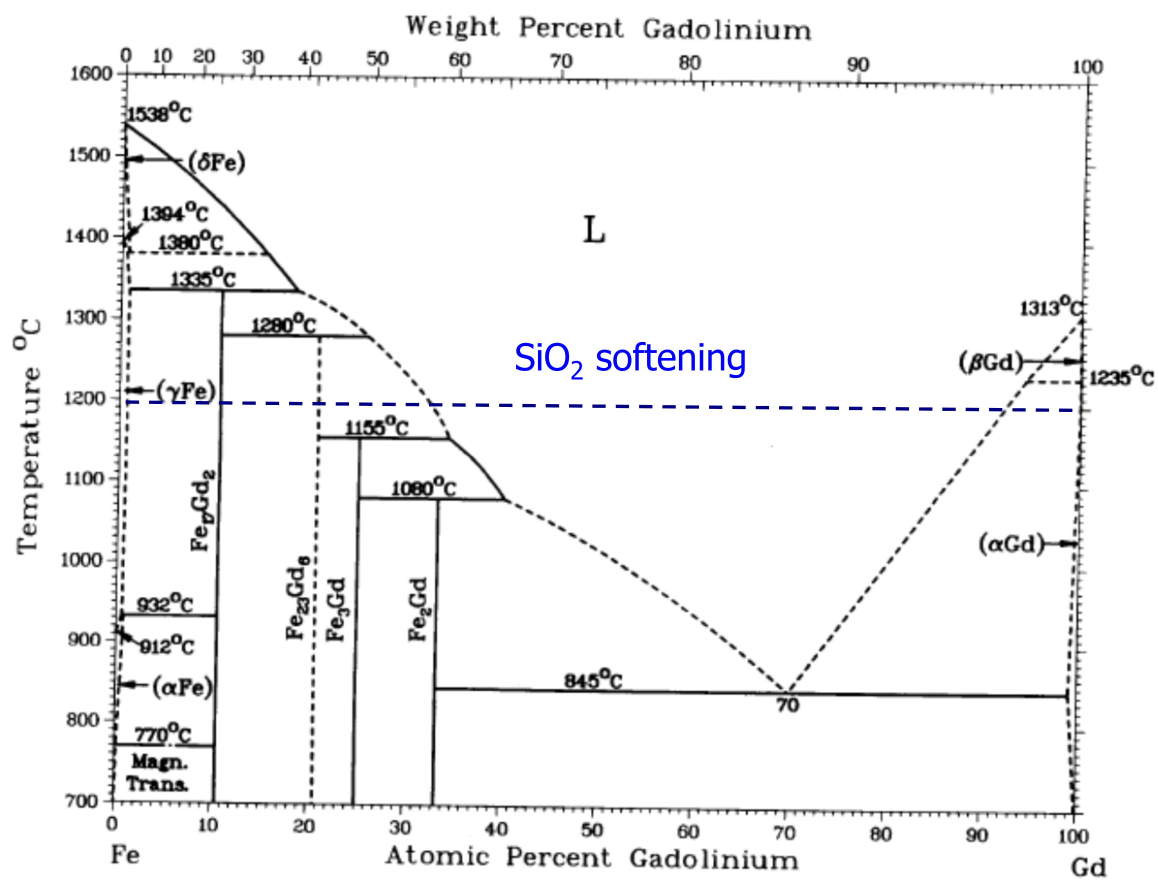


Heat to 1200 C and cool

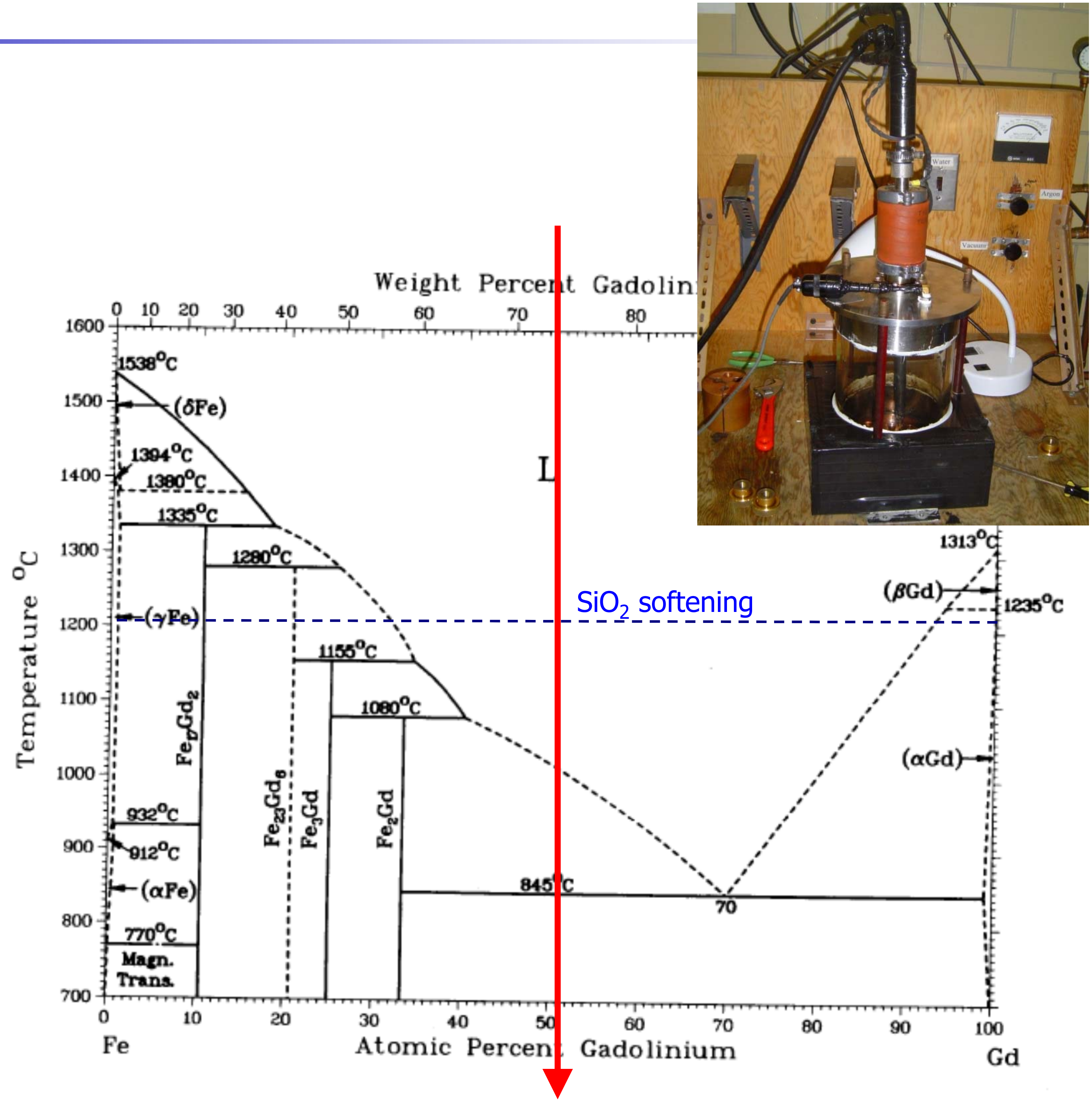


Let's try to grow $GdFe_2$. This presents several problems. The first is the fact that both elements are relatively high melters. If we just put Gd and Fe in a crucible and heated to 1200 C they would not react (surface area of contact matters).



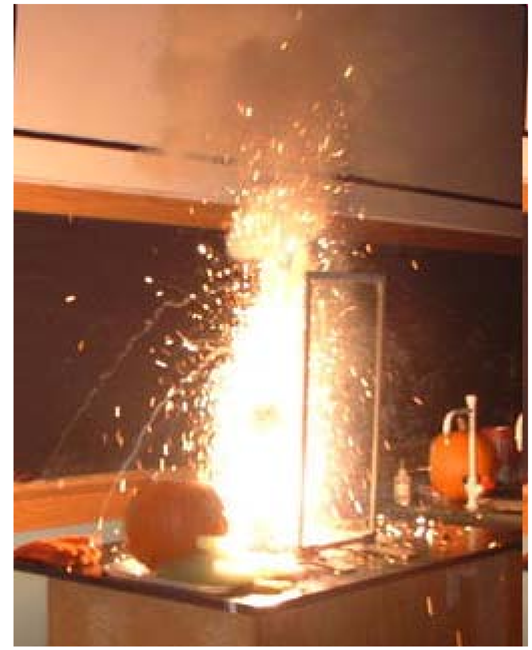


To solve this problem, an arc-melter can be used to pre-alloy (essentially finely mix through quench cooling) the elements to allow growth below 1200 C



Second problem:

Lots of Gd will attack Al_2O_3 via a thermite-type reaction



Classic thermite reaction: $Fe_2O_3 + 2Al \rightarrow Al_2O_3 + 2Fe + \Delta$ (heat)

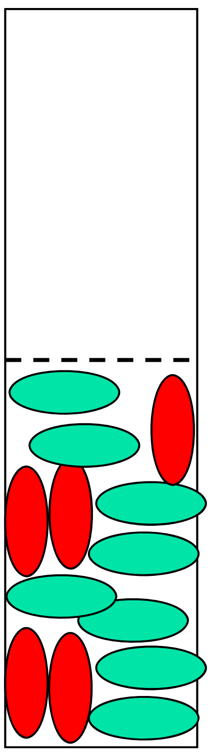
Or in this case, Gd reacts with Al_2O_3.....

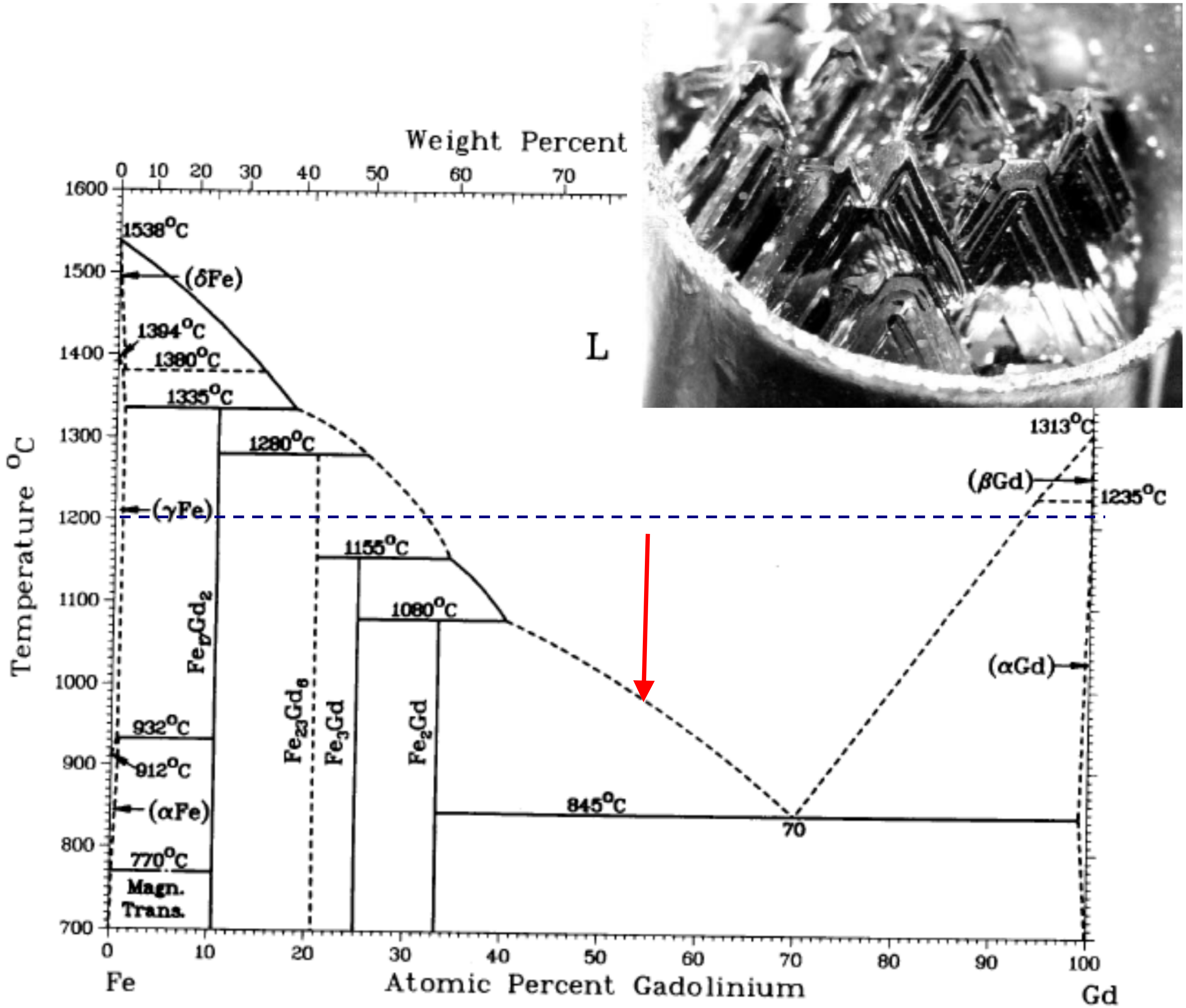
$Al_2O_3 + 2Gd \rightarrow Gd_2O_3 + 2Al + \Delta$ (heat)

(Results in damaged / leaking crucible as well as contaminated and depleted melt.)

Lots of Gd which attacks Al_2O_3 via the thermite reaction

We solved this by developing a 3-cap Ta crucible.





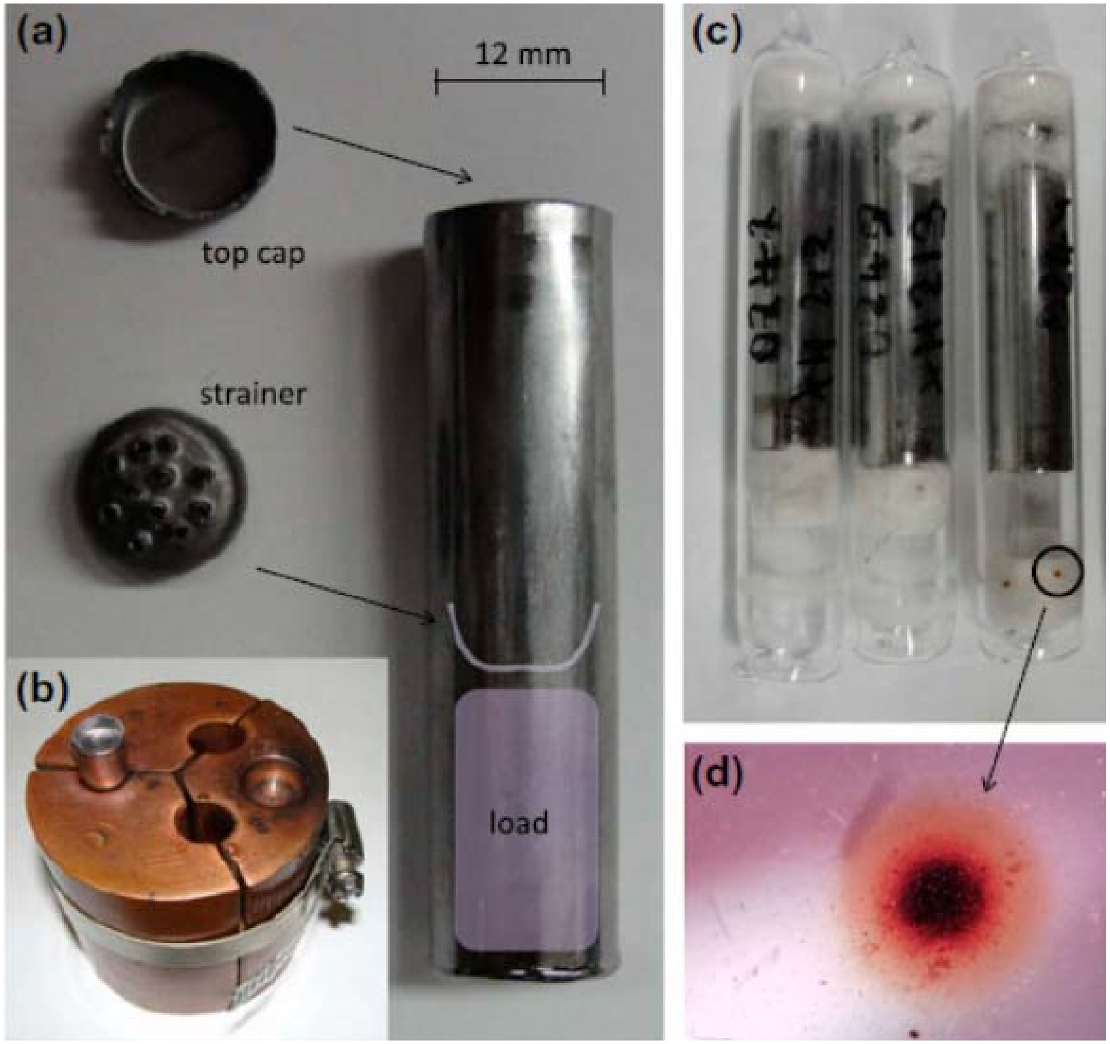
Journal of Crystal Growth 225 (2001) 155–161

Single crystal growth from light, volatile and reactive materials using lithium and calcium flux

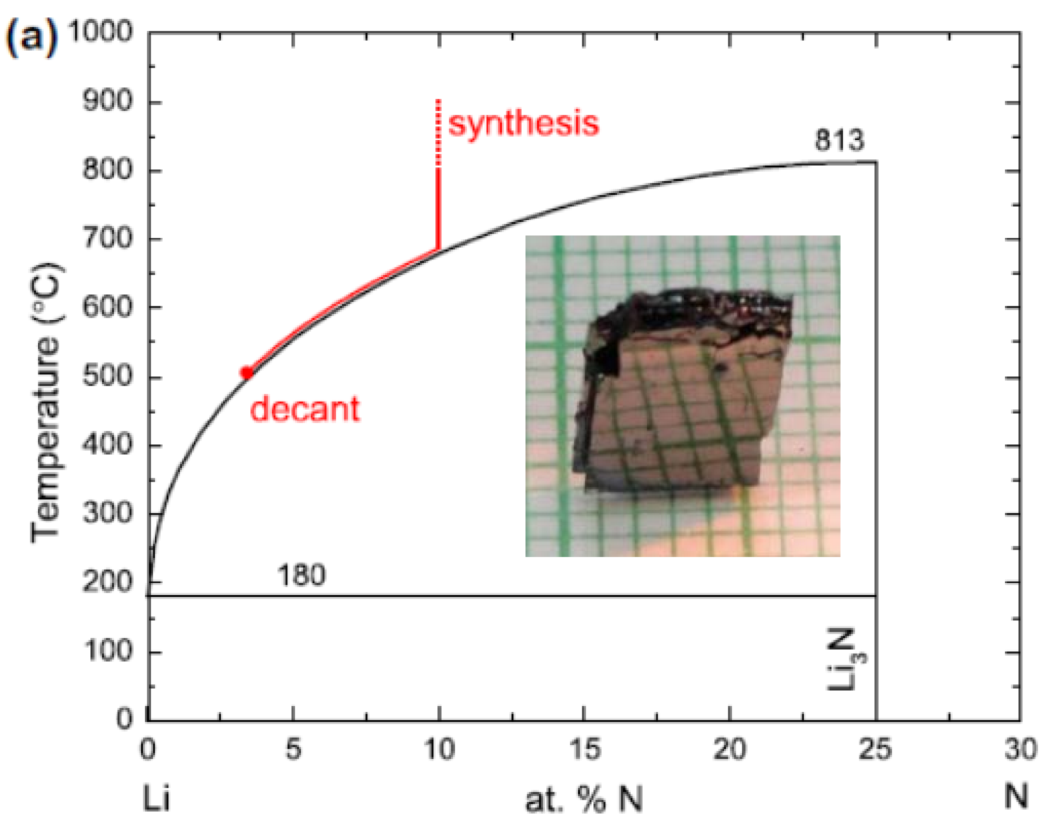
A. Jesche[a*] and P.C. Canfield[ab]

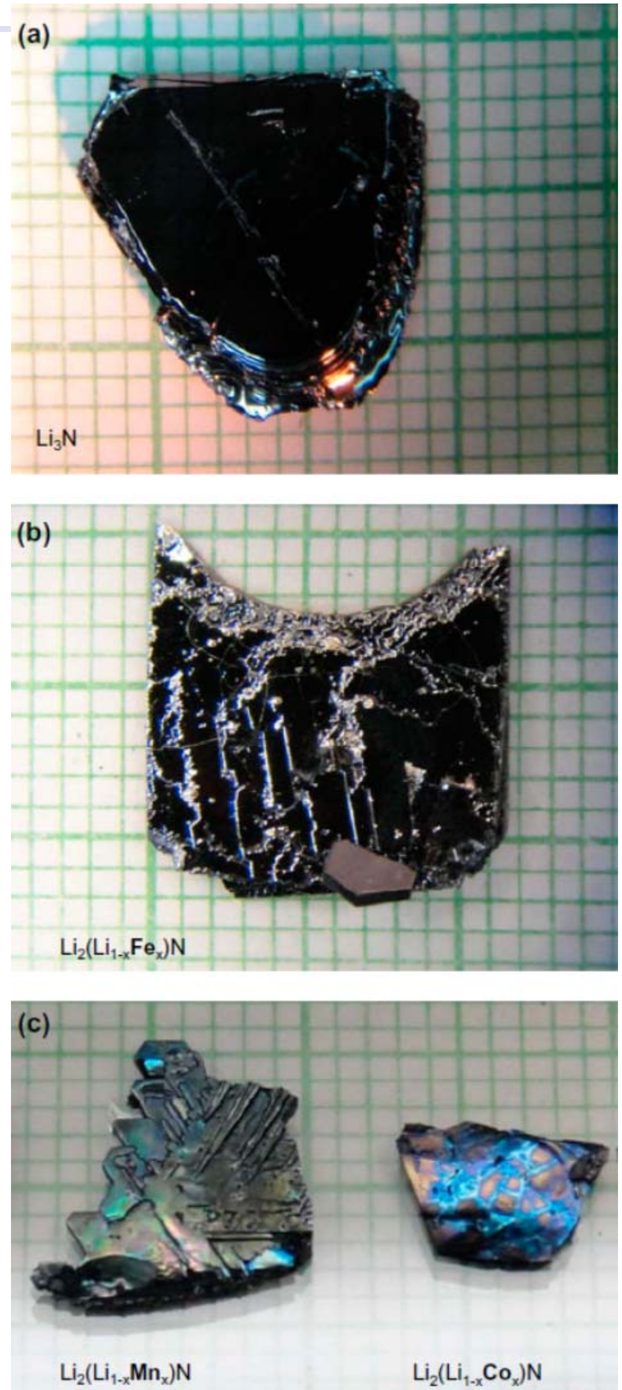
Philosophical Magazine, 2014
Vol. 94, No. 21, 2372–2402, http://dx.doi.org/10.1080/14786435.2014.913114

We extended the use of Ta-3-cap crucibles from R-rich melt to Mg-based melts all the way to Li- and Ca-base melts. This allowed us to handle a wide variety of reactive elements for solution growth.



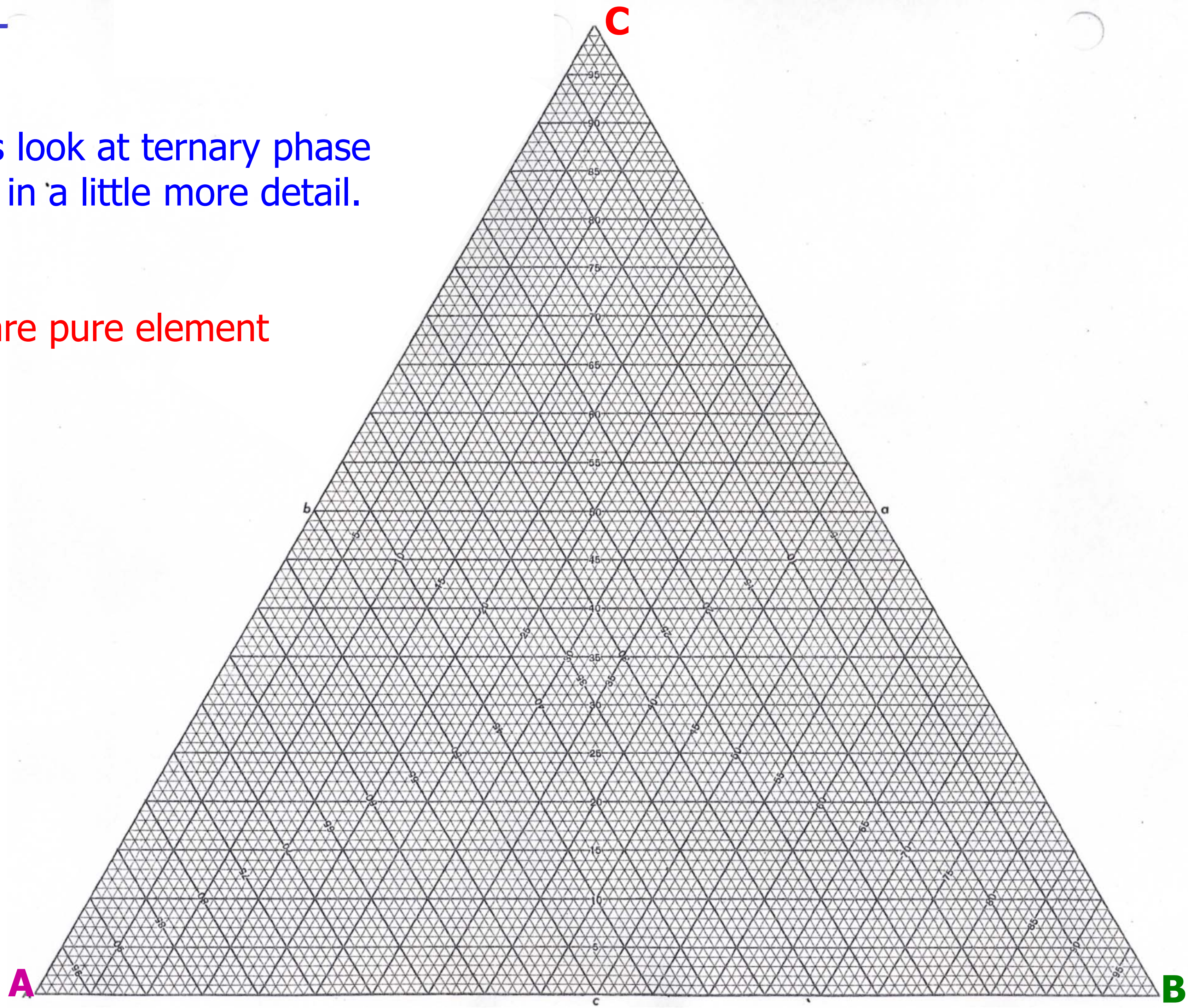
For example, we grew Li_3N out of excess Li and then were also able to study $(Li_{1-x}T_x)Li_2N$ compounds for T = Mn, Fe, Co, Ni.

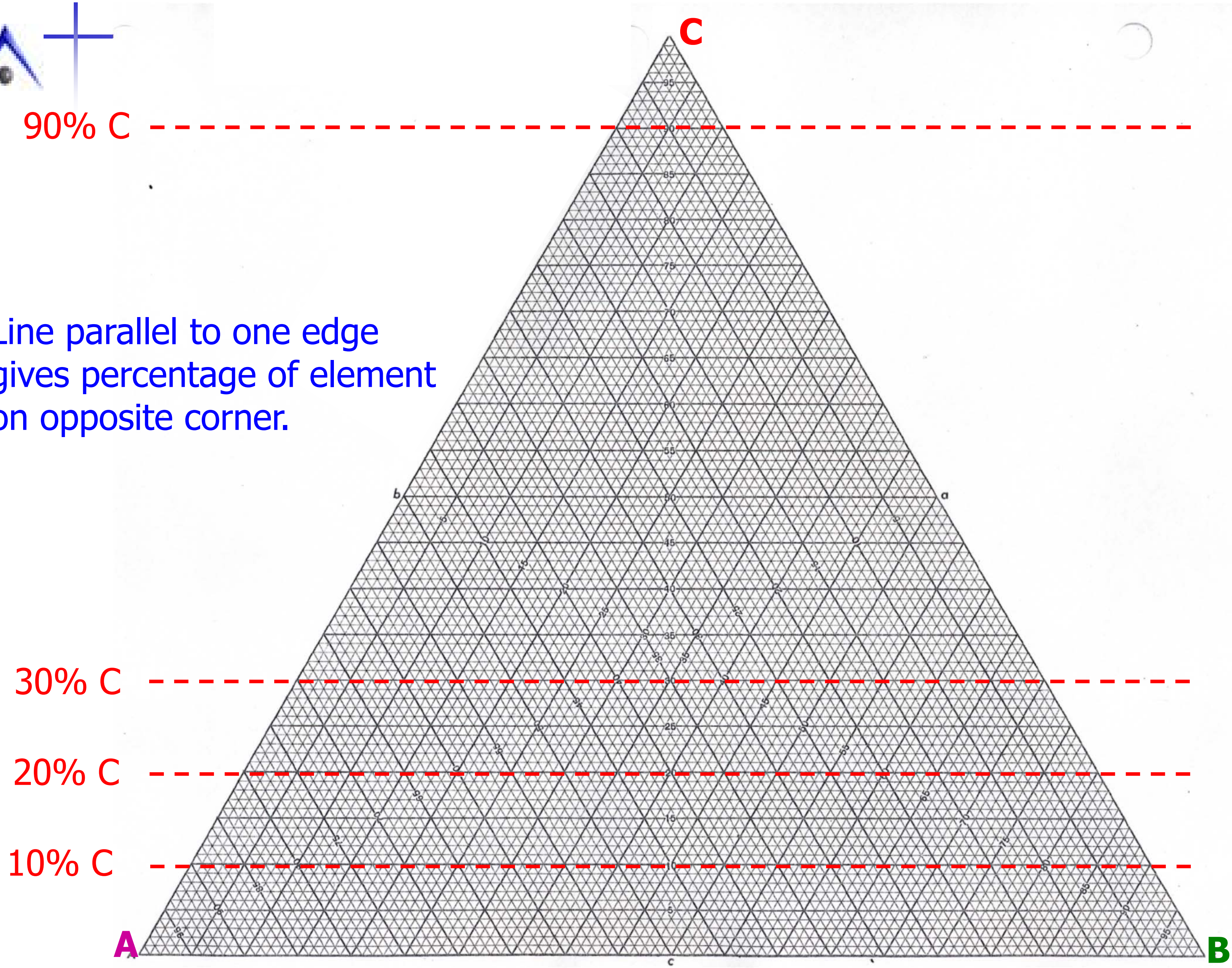




Now, let's look at ternary phase diagrams in a little more detail.

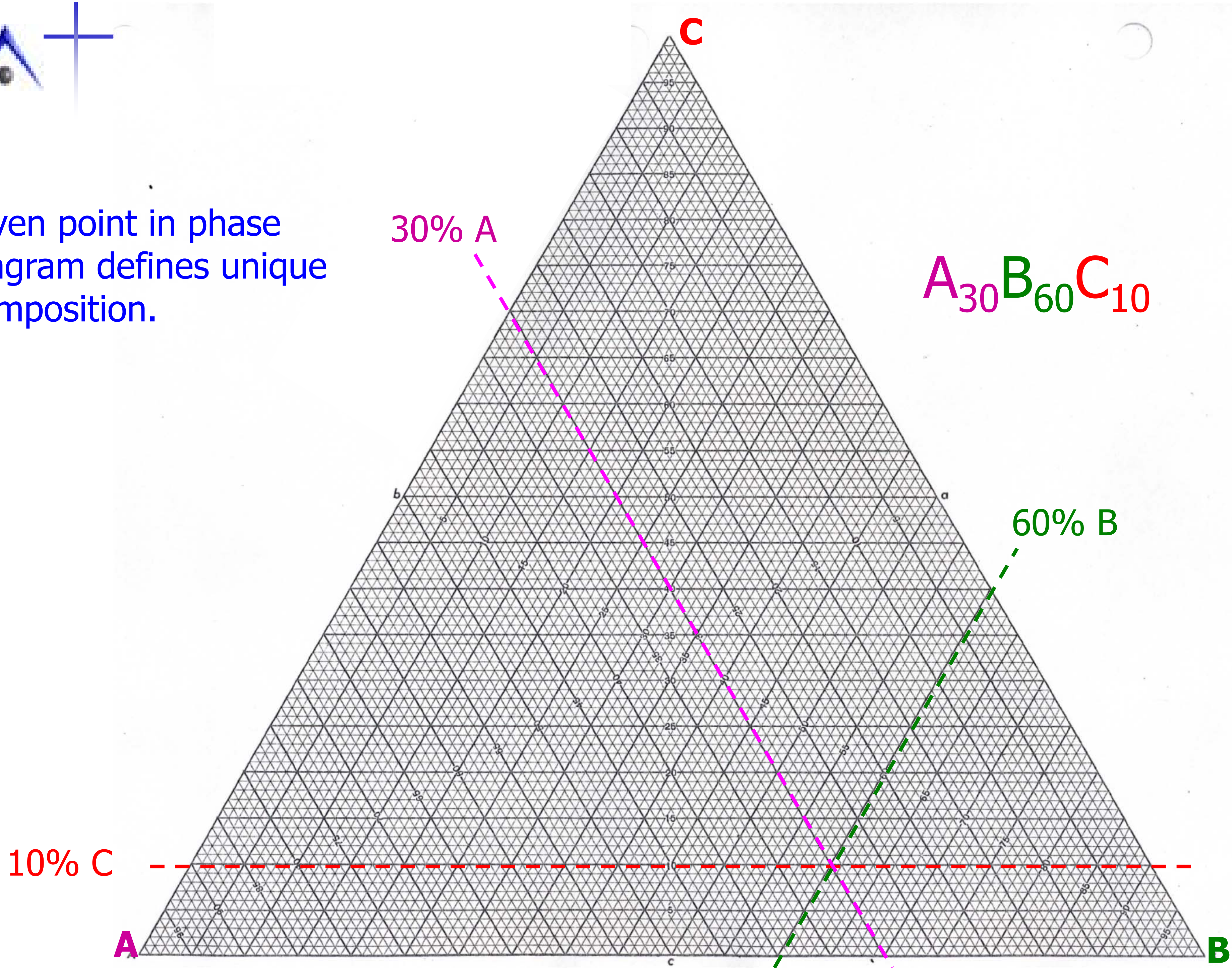
Corners are pure element



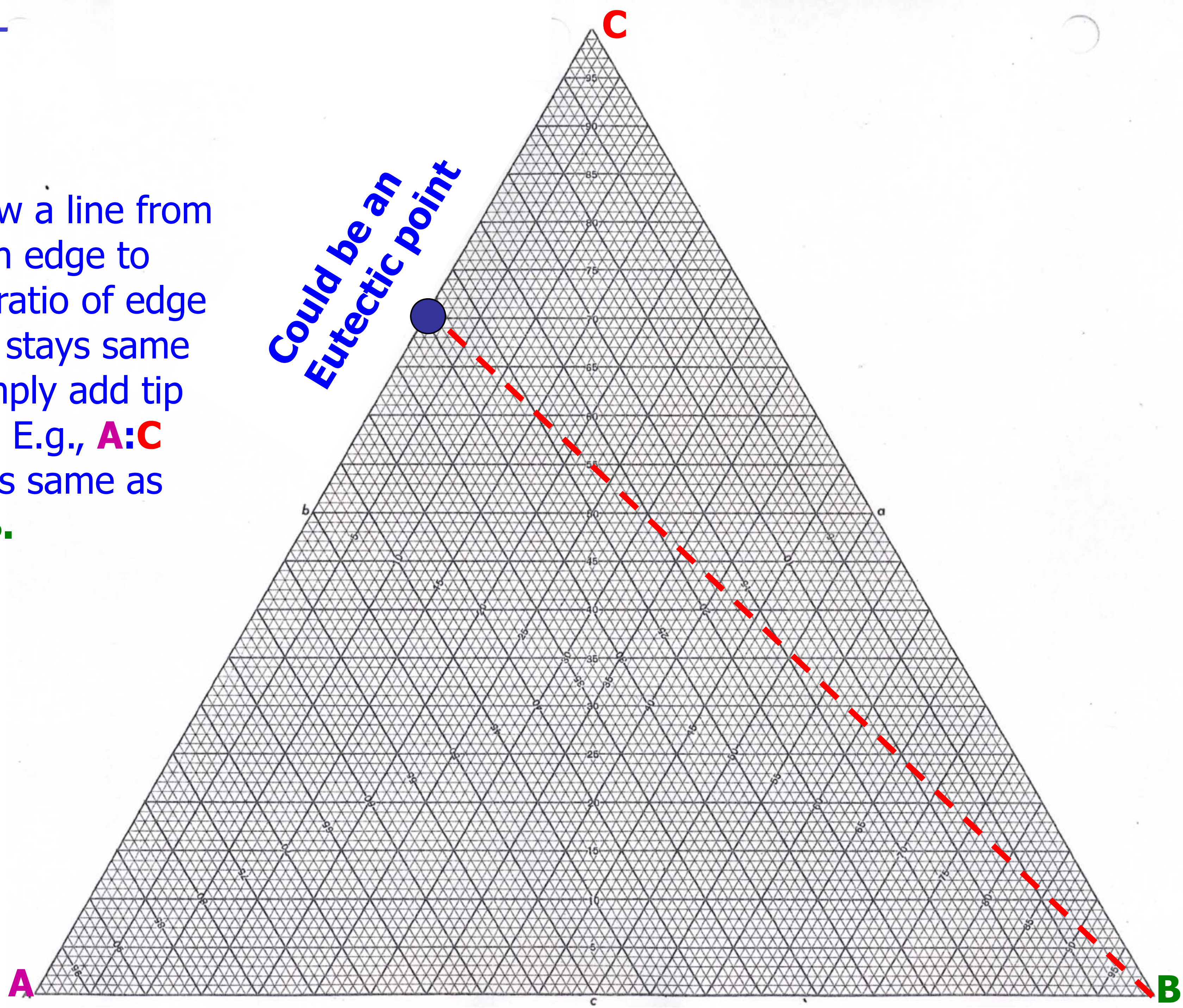


Line parallel to one edge gives percentage of element on opposite corner.

Given point in phase diagram defines unique composition.



If we draw a line from a point on edge to tip, then ratio of edge elements stays same as we simply add tip element. E.g., **A:C** ratio stays same as we add **B.**

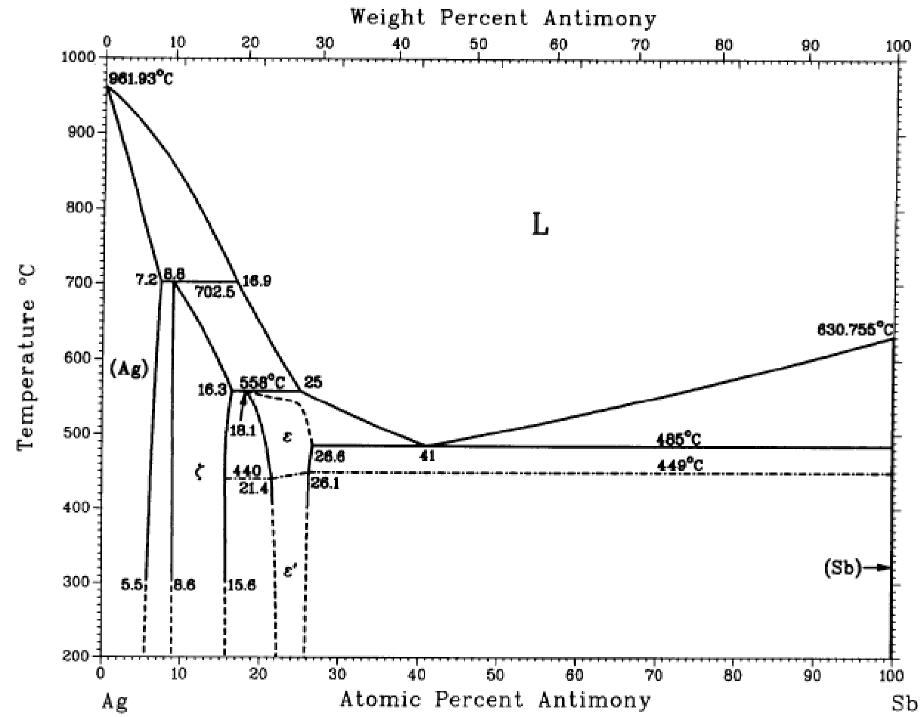


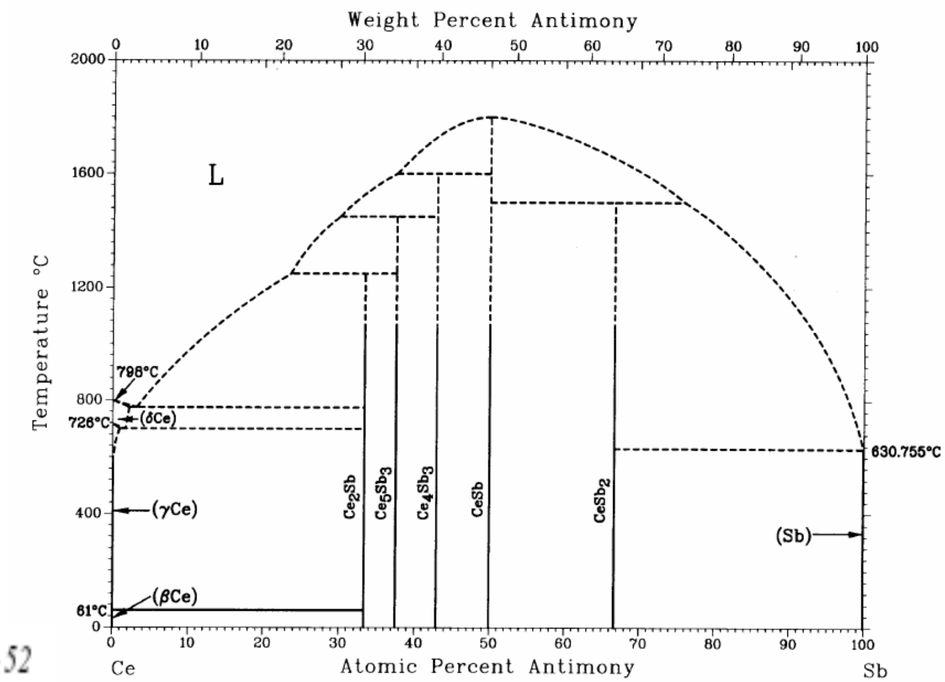
Now for some ternary compounds.

$RAgSb_2$ to start

$RAgSb_2$ compounds can be grown out of excess Sb.

This is similar in spirit to growing $CeSb_2$ out of excess Sb: we are growing out of an excess of one of the constituent elements

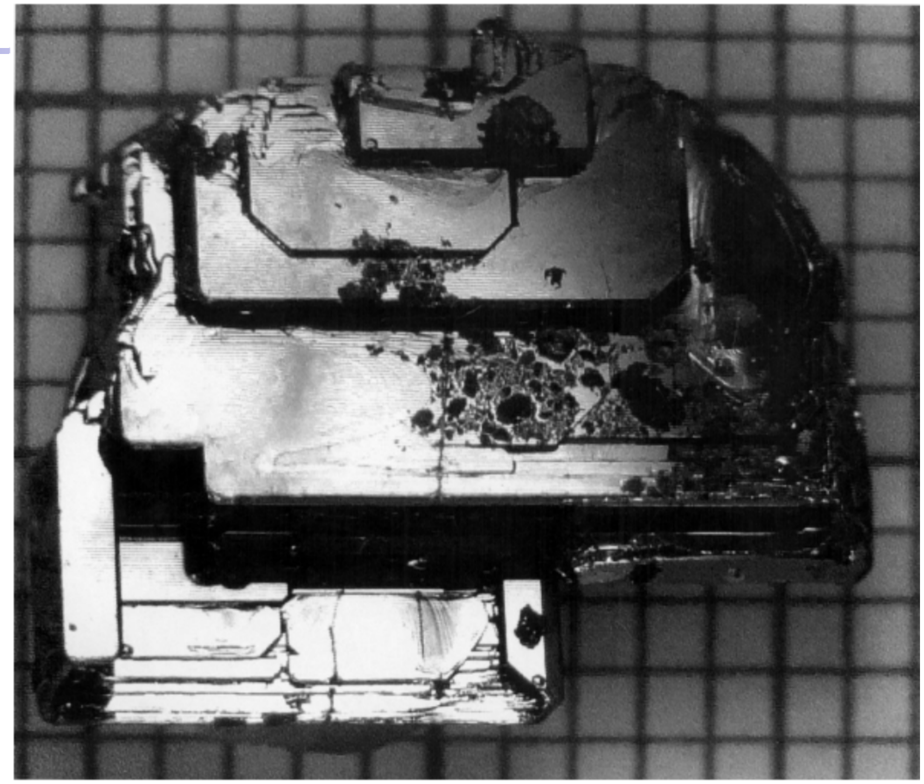


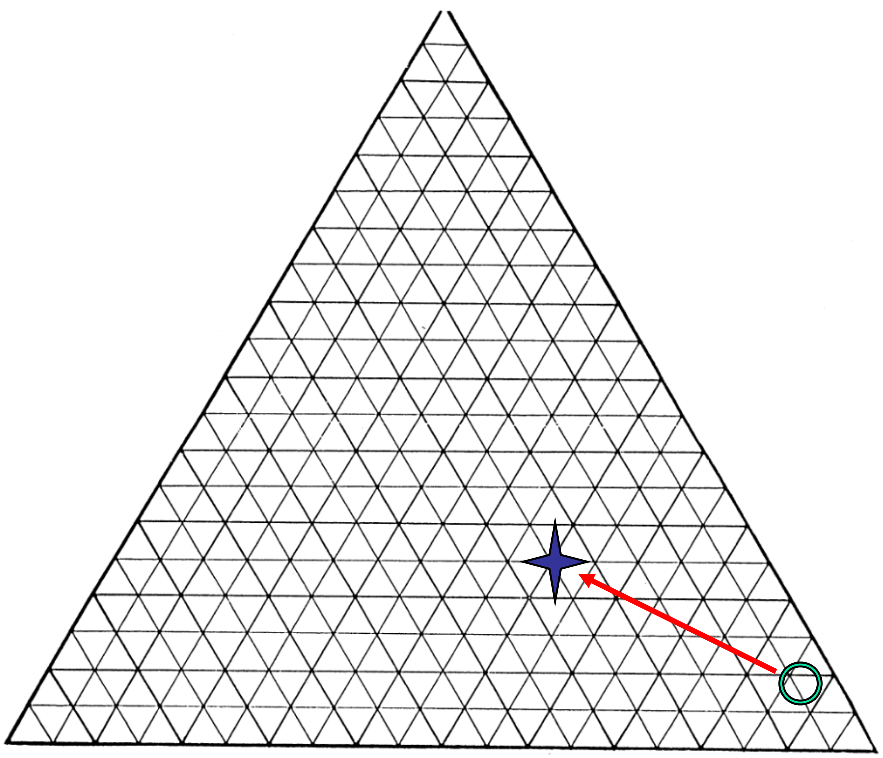


K.D. Myers et al. / Journal of Magnetism and Magnetic Materials 205 (1999) 27–52

K.D. Myers et al. / Journal of Magnetism and Magnetic Materials 205 (1999) 27–52

$CeAgSb_2$ ($Ce_{25}Ag_{25}Sb_{50}$) can be grown from a melt with initial stoichiometry of $Ce_4Ag_9Sb_{87}$. Same works for R = La, Ce, Pr, Nd…





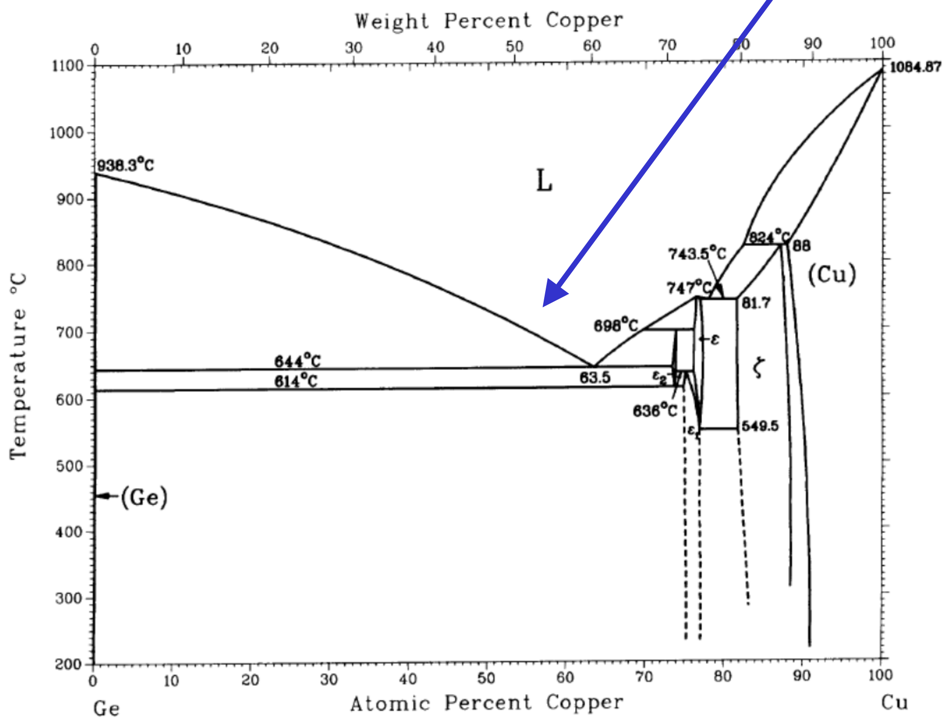
This growth can be place in Al_2O_3 and sealed in silica. Temperature profile is:

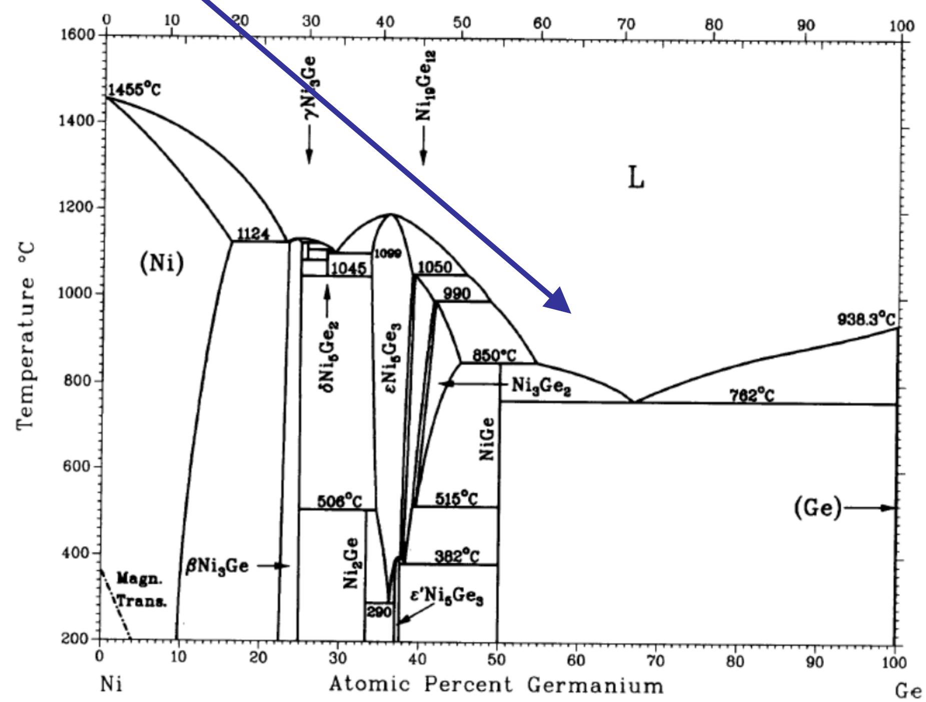
$$1200\text{ C} \xrightarrow{\text{120 hours}} 670\text{ C}$$

Now ternary growths out of eutectics:

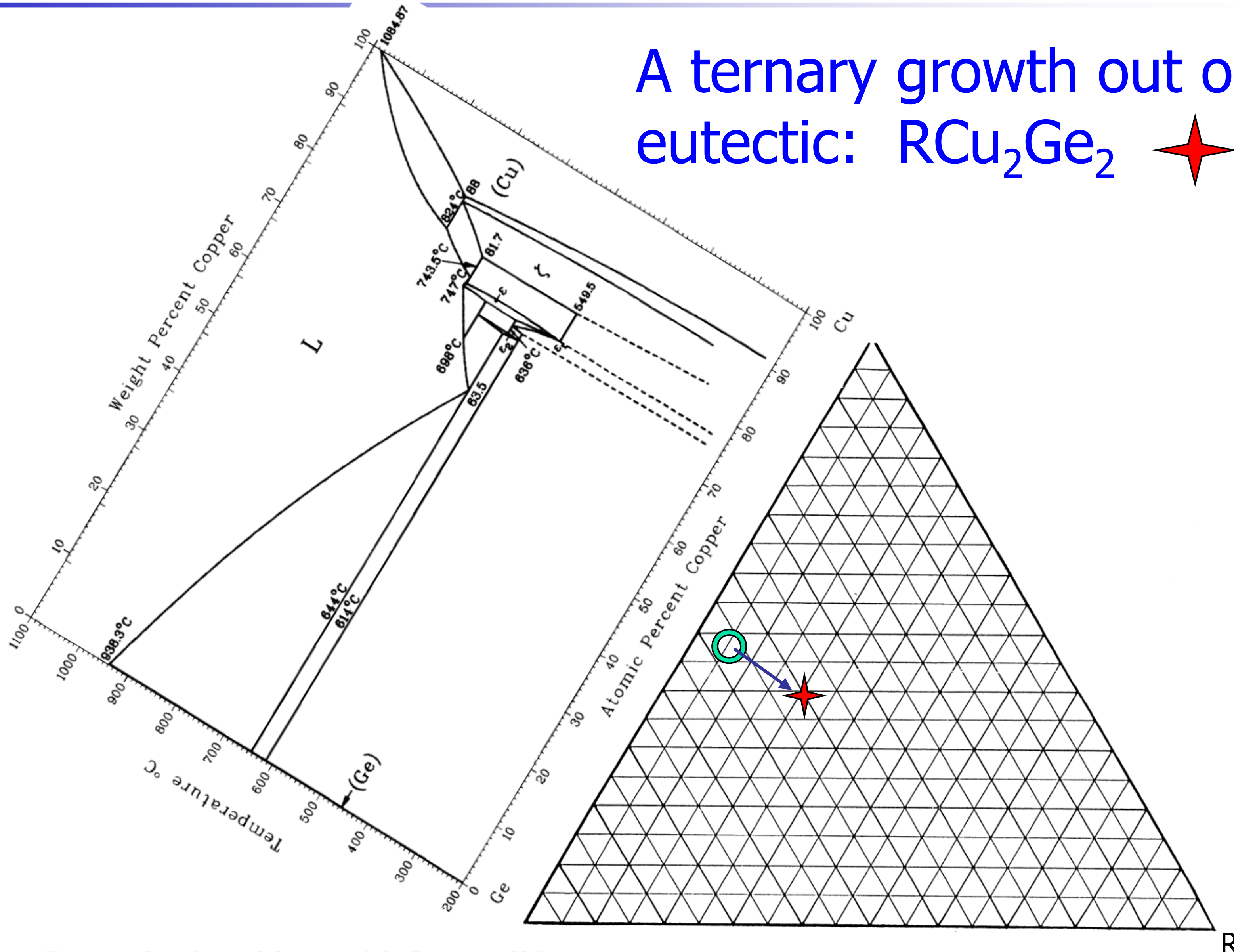
RNi_2Ge_2 and RCu_2Ge_2 series.

Both the Ge-Cu and the Ge-Ni binaries have low, broad eutectic valleys.



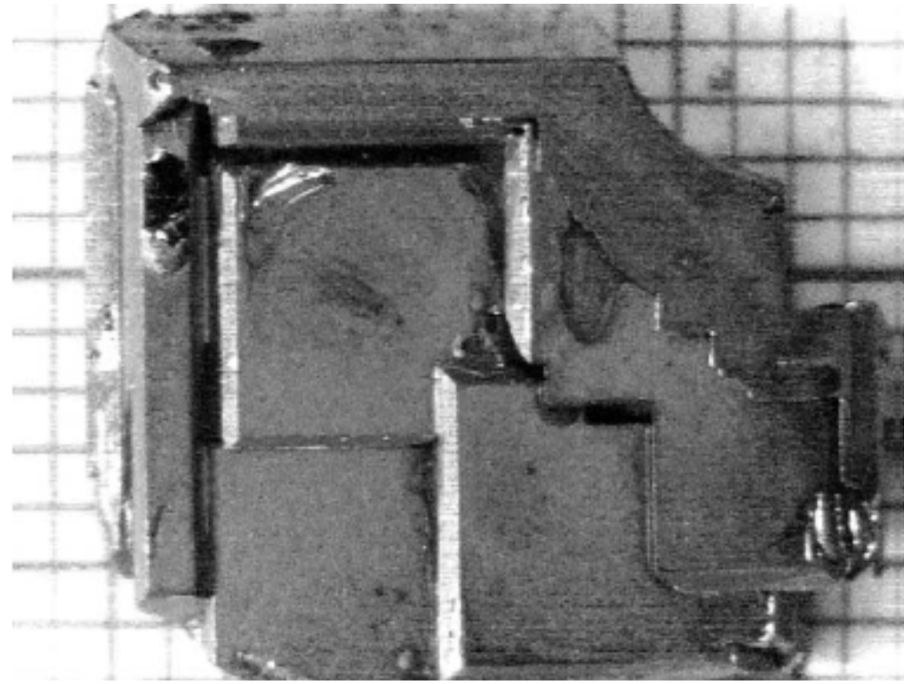


A ternary growth out of an eutectic: RCu_2Ge_2



$Ce_{0.05}Cu_{0.475}Ge_{0.475}$ is placed in an Al_2O_3 crucible, sealed in a quartz ampule and heated to 1190°C. The ampule is cooled to 825°C over 200 h and then the excess liquid is decanted. The resulting crystal

$CeCu_2Ge_2$ (m = 2 g)



Grown in a 5 ml Al_2O_3 crucible, sealed in silica

200 hours

1190 C $\longrightarrow$ 825 C

Here is a 1992 summary of binary and ternary samples that I had grown out of binary, ternary and quaternary melts.

This is a very powerful technique for exploratory growth. Over about two (plus) decades my group has made close to 10,000 growths similar to the ones I have been describing.

Growth of single crystals from metallic fluxes

Philosophical Magazine B, 1992, Vol. 65, No. 6, 1117–1123

Crystal growth from metallic flux.

Crystals	Flux	Dilution[a] (at.%)	Temperature (°C)	Comments
RB_4	Al	R, 0·2	1450–700	R ≡ Sm, Gd–Lu, also U, Th
$YbAlB_4$	Al	Yb, 1·0	1450–700	
RB_6	Al	R, 0·2	1450–700	R ≡ La–Eu, Yb, Ca, Ba, Sr, Np, Am
RBe_{13}	Al	R, 5·0	1250–700	R ≡ La–Lu, Y, U, Th BeO crucible
RAl_3	Al	Yb, 0	1200–660	R ≡ Yb, Lu, Y, Sc
TiB_2	Al	Ti, 2·0	1440–800	No spin, NaOH etch
$CeSi_{2-x}$	Al	Ce, 5·0	1150–800	No spin, NaOH etch
UAl_3	Bi	U, 2·0	1150–700	
UPt_3	Bi	U, 7·0	1250–800	BeO crucible
YPd	Bi	Y, 10	1175–600	
RBiPt	Bi	Ho, 6·0; Yb, 10; Lu, 3·0	1150–500	R ≡ Nd–Lu
$R_3Bi_4Pt_3$	Bi	Ce, 13	1150–500	R ≡ La–Pr
RBi_2	Bi	Ce, 10	800–400	R ≡ La, Ce, Pr, Yb
R_2Bi	Ce	Bi, 15	1150–900	R ≡ La, Ce, Ta crucible
Ce_2Sb	Ce	Sb, 12	1150–900	Ta crucible
$CeFe_2$	Ce	Fe, 45	1100–700	Ta crucible
RSb	Ga	Ce, 5·0	1150–650	R ≡ La–Nd
$R_2Pt_4Ga_8$	Ga	Ce, 1·0	1100–500	R ≡ La–Nd, Sm, Gd, etc.
$CeCu_2Ge_2$	In	Ce, 3·0	1175–750	Plates 2 mm × 2 mm × 0·2 mm
$CeNi_2Ge_2$	In	Ce, 3·0	1175–700	Plates 2 mm × 2 mm × 0·2 mm
$YbCu_2Si_2$	In	Yb, 4·0	1150–600	Plates 2 mm × 2 mm × 0·2 mm
RPb_3	Pb	Ce, 10	1100–800	R ≡ La, Ce
RPbPt	Pb	Ce, 7·0	1150–500	R ≡ La, Ce
RBiPt	Pb	Ce, 7·0	1150–500	R ≡ La, Ce, Pr
$YbCu_2Si_2$	Sn	Yb, 3·0	1150–700	R ≡ La, Ce, Pr
TiNiSn	Sn	Ti, 9·0	1150–600	Pyramidal
MnSnNi	Sn	Mn, 10	1150–450	Pyramidal
RSb	Sn	Ce, 5·0	1150–750	R ≡ La–Nd
RSb_2	Sb	Ce, 10	1175–750	R ≡ La, Ce
$U_3Sb_4Pt_3$	Sb	U, 8·0	1150–750	
$PtSb_2$	Sb	Pt, 10	1150–750	

[a] All materials in this table are dissolved in the flux stoichiometrically. The values shown for dilution are the amounts of one of the crystal components with respect to the flux.

Since 1992 we have made on the order of 10,000 more growths and published several "growth papers" in Philosophical Magazine

PHILOSOPHICAL MAGAZINE B, 1992, VOL. 65, NO. 6, 1117–1123

Growth of single crystals from metallic fluxes

Philosophical Magazine
Vol. 92, Nos. 19–21, 1–21 July 2012, 2436–2447

Development of viable solutions for the synthesis of sulfur bearing single crystals

Philosophical Magazine
Vol. 92, Nos. 19–21, 1–21 July 2012, 2448–2457

Growing intermetallic single crystals using *in situ* decanting

Philosophical Magazine, 2014
Vol. 94, No. 21, 2372–2402, http://dx.doi.org/10.1080/14786435.2014.913114

Single crystal growth from light, volatile and reactive materials using lithium and calcium flux

PHILOSOPHICAL MAGAZINE, 2016
VOL. 96, NO. 1, 84–92
http://dx.doi.org/10.1080/14786435.2015.1122248

Use of frit-disc crucibles for routine and exploratory solution growth of single crystalline samples

Summary:

I hope that some of you now simply want to get into the lab a play around with growth.

You are NOT limited to the four samples that your Professor found in a drawer.

All Physics does not *have* to be found in Si.

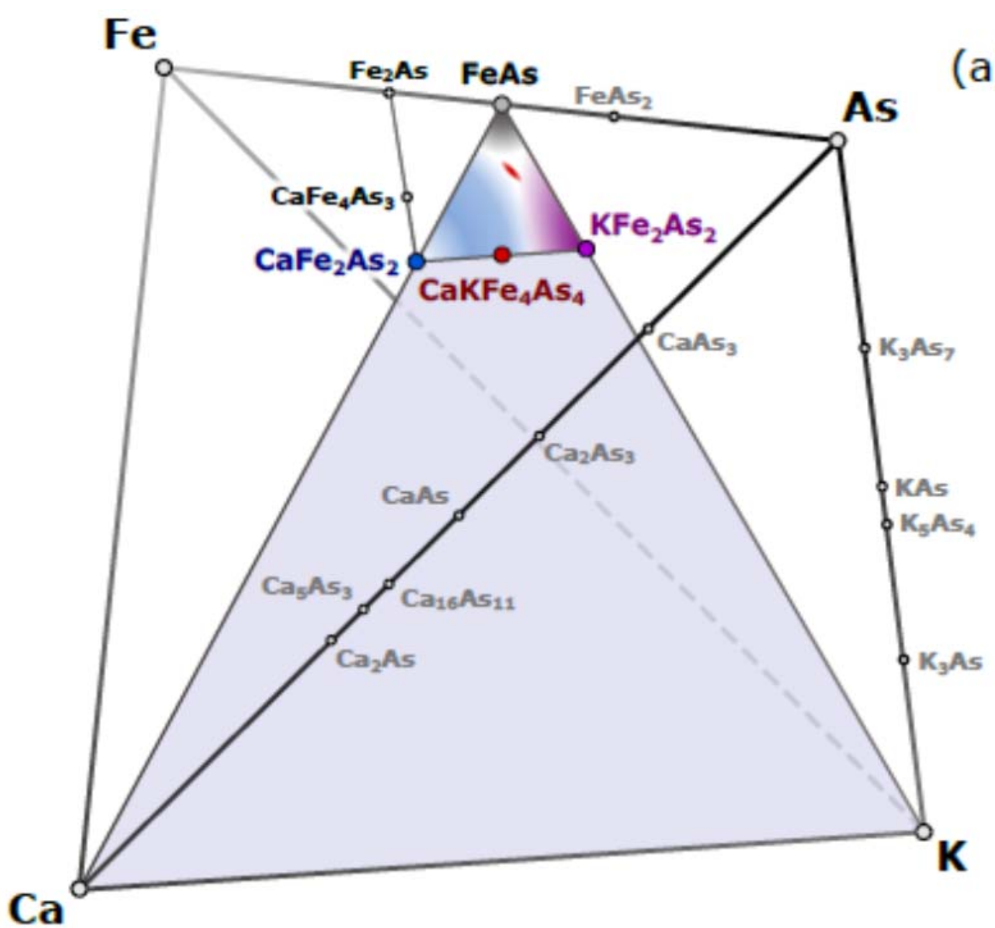
You can try to discover, design, and make crystals that will allow you to pursue the specific science that interest YOU.


Questions?

PHYSICAL REVIEW MATERIALS 1, 013401 (2017)

Optimization of the crystal growth of the superconductor $CaKFe_4As_4$ from solution in the FeAs-$CaFe_2As_2$-KFe_2As_2 system

W. R. Meier, T. Kong,* S. L. Bud'ko, and P. C. Canfield†
Ames Laboratory US DOE and Department of Physics and Astronomy, Iowa State University, Ames, Iowa 50011, USA
(Received 28 April 2017; published 19 June 2017)



This is a true quaternary growth!!

PHYSICS 590B FALL 2018 SCHEDULE **MWF 1:10 – 2:00 p.m.** **Room 38 Physics Hall**

Week 1 / Aug 20 Intro (Canfield), Aug 22, 24 Measuring of Temperature (Prozorov)

Week 2/ Aug 27, 29 Cryogens: generation and handling (Bud'ko) Aug 31 Low pressure generation and gauging (Kaminski)

Week 3 / Sept. 5, 7 Low pressure generation and gauging (Kaminski)

Week 4 / Sept 10, 12, 14 X-ray and Neutron Generation (in-house as well as facilities) (Goldman/McQueeney/Kreyssig)

Week 5/ Sept 17, 19, 21 Elastic X-ray and neutron scattering (Goldman/McQueeney/Kreyssig)

Week 6/Sept 24, 26, 28 E and B field generation (Kaminski and Bud'ko)

Week 7 / Oct 1, 3, 5 First sets of 10 plus 6 minute talks from students.

Week 8 / Oct 8, 10, 12 Magnetization measurements (d.c.) (Prozorov)

Week 9 / Oct 15, 17, 19 Magnetization measurements (a.c., magneto optics, NV) (Prozorov)

Week 10 / Oct 22, 24, 26 Specific heat and scanning calorimetry measurements (Bud'ko/ Dennis)

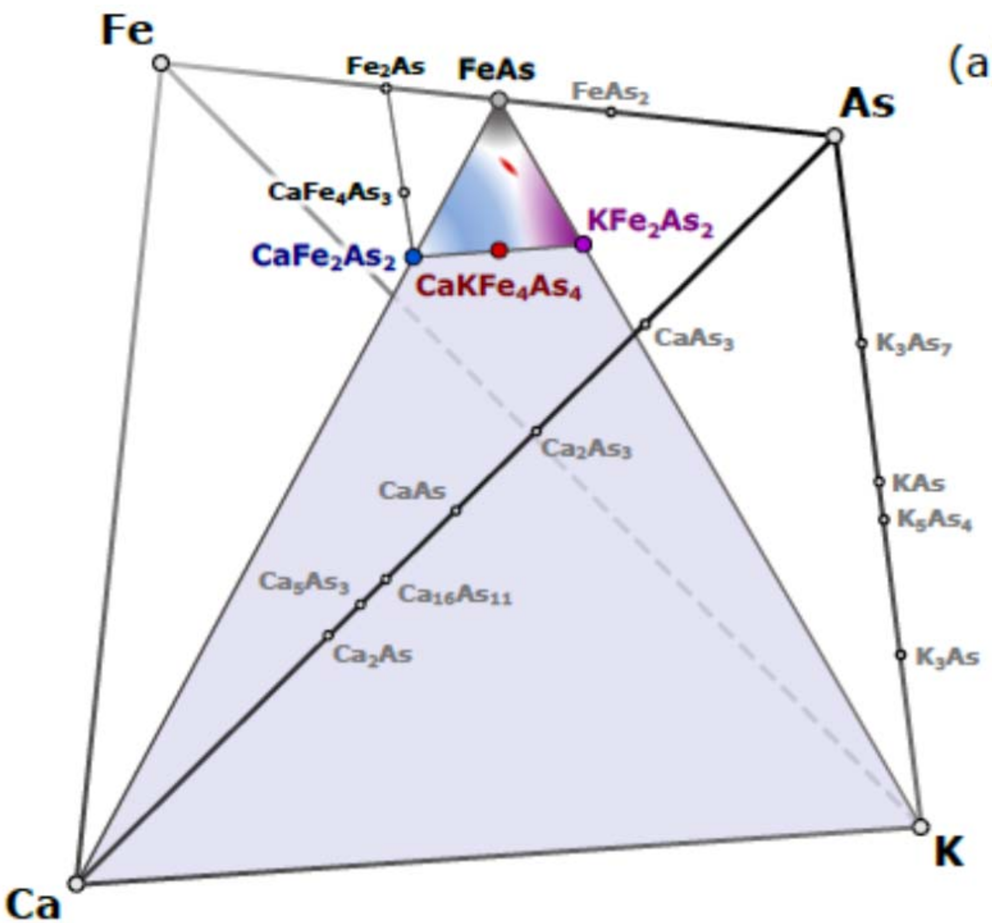
Week 11 / Oct 29, 31, Nov 2 Compositional phase diagrams, how to read them and how to make them (W. Meier)

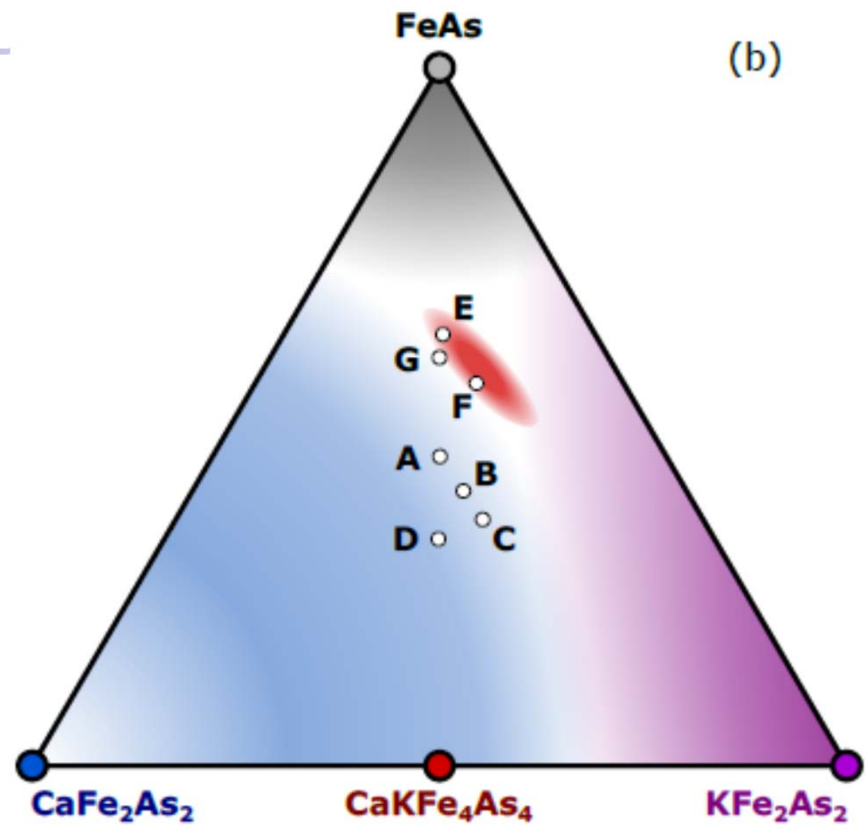
Week 12 / Nov 5, 7, 9 Elemental Analysis and e-beam (Straszheim / Kramer)

Week 13 / Nov 12, 14 High pressure generation, gauging and measurements (Bud'ko) Nov 16 Specific heat under pressure (Gati)

Week 14 / Nov 26, 28, 30 Electrical resistivity measurements (Tanatar)

We can start with the formal tetrahedron

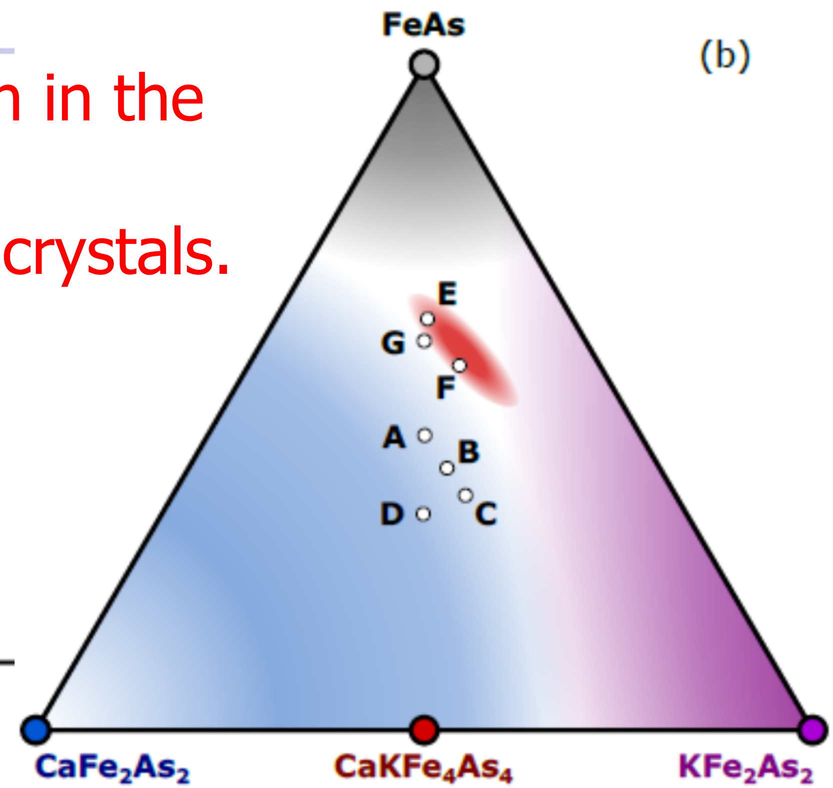




And then make a "simplifying" pseudo-ternary cut.

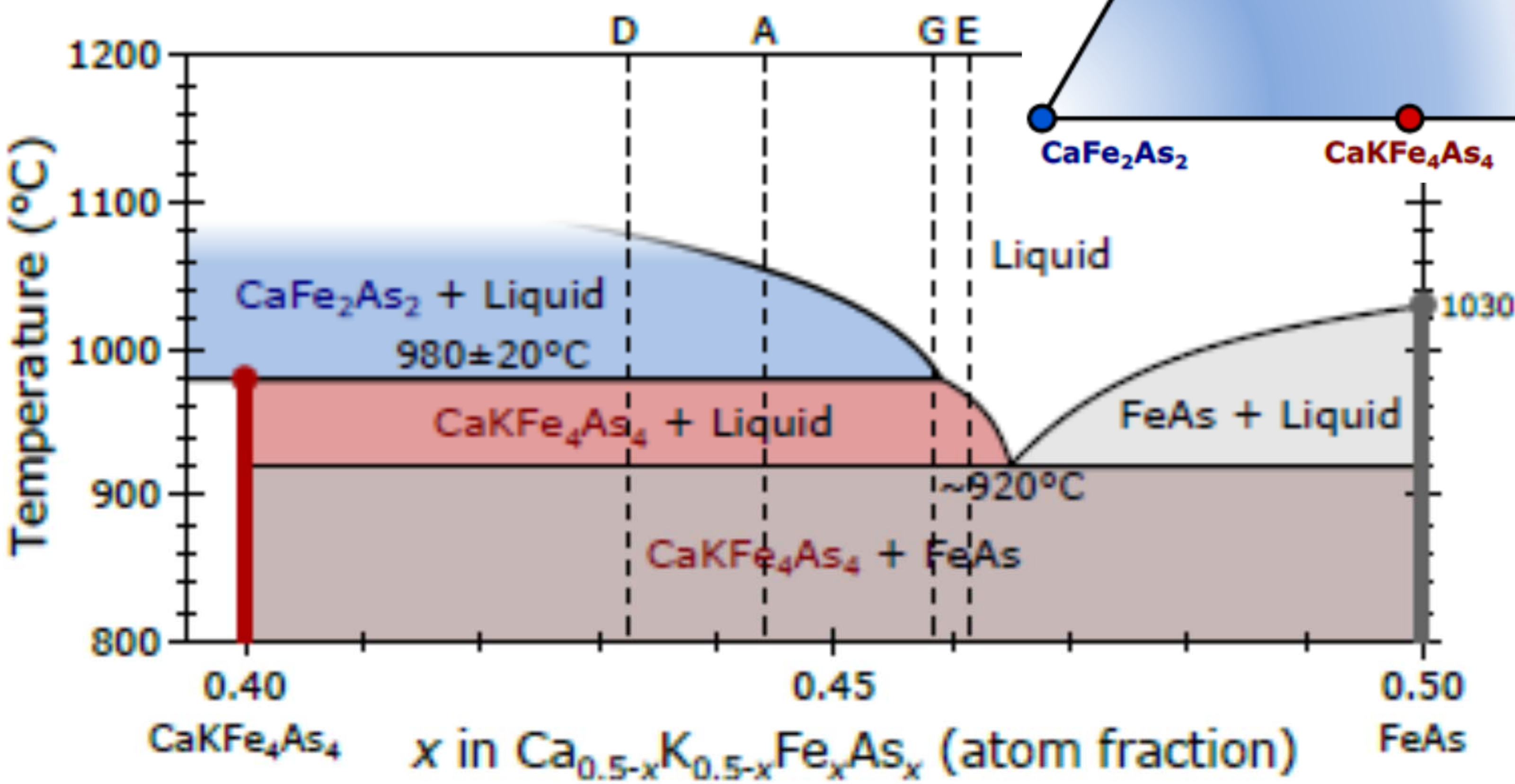
PHYSICAL REVIEW MATERIALS 1, 013401 (2017)

We could identify the red region in the pseudo-ternary cut as primary solidification for the $CaKFe_4As_4$ crystals.



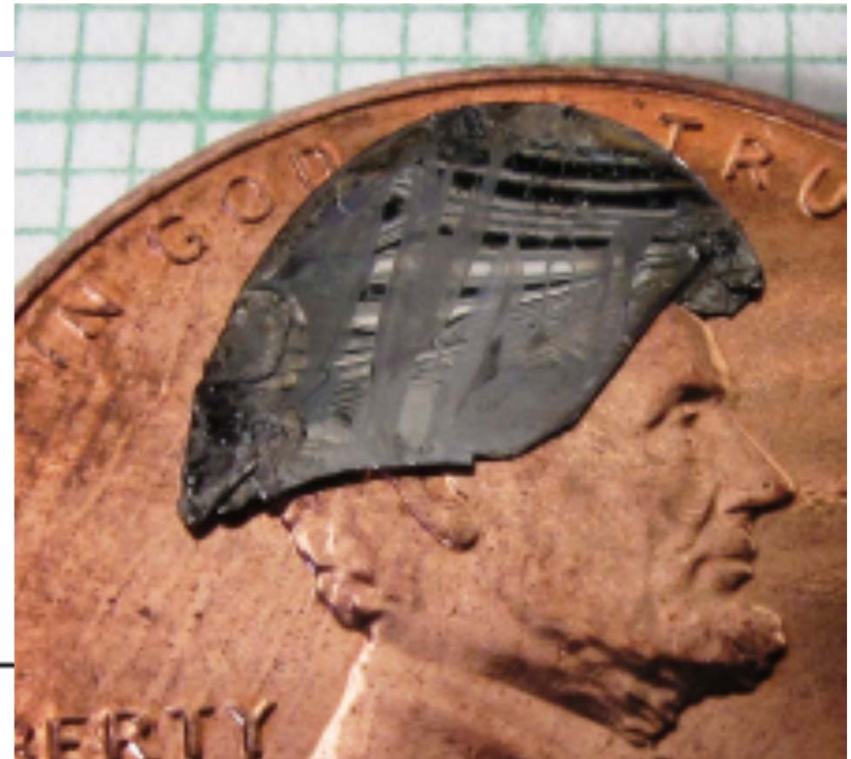
This can be seen in the pseudo-binary cut below.

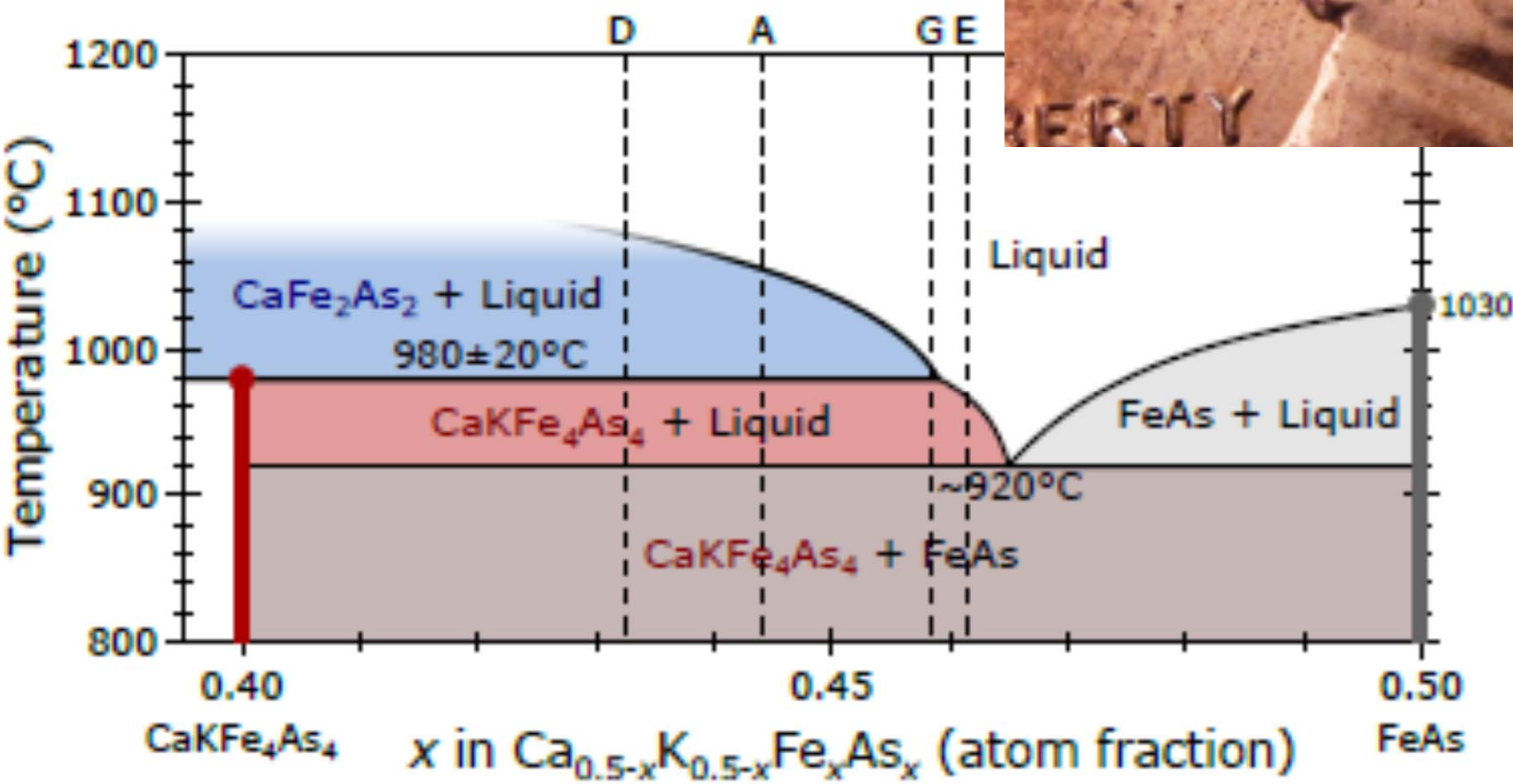
PHYSICAL REVIEW MATERIALS 1, 013401 (2017)



PHYSICAL REVIEW MATERIALS 1, 013401 (2017)

By hitting this very limited region of primary solidification we can grow sizable $CaKFe_4As_4$ crystals.





PHYSICS 590B FALL 2018 SCHEDULE **MWF 1:10 – 2:00 p.m.** **Room 38 Physics Hall**

Week 1 / Aug 20 Intro (Canfield), Aug 22, 24 Measuring of Temperature (Prozorov)

Week 2/ Aug 27, 29 Cryogens: generation and handling (Bud'ko) Aug 31 Low pressure generation and gauging (Kaminski)

Week 3 / Sept. 5, 7 Low pressure generation and gauging (Kaminski)

Week 4 / Sept 10, 12, 14 X-ray and Neutron Generation (in-house as well as facilities) (Goldman/McQueeney/Kreyssig)

Week 5/ Sept 17, 19, 21 Elastic X-ray and neutron scattering (Goldman/McQueeney/Kreyssig)

Week 6/Sept 24, 26, 28 E and B field generation (Kaminski and Bud'ko)

Week 7 / Oct 1, 3, 5 First sets of 10 plus 6 minute talks from students.

Week 8 / Oct 8, 10, 12 Magnetization measurements (d.c.) (Prozorov)

Week 9 / Oct 15, 17, 19 Magnetization measurements (a.c., magneto optics, NV) (Prozorov)

Week 10 / Oct 22, 24, 26 Specific heat and scanning calorimetry measurements (Bud'ko/ Dennis)

Week 11 / Oct 29, 31, Nov 2 Compositional phase diagrams, how to read them and how to make them (W. Meier)

Week 12 / Nov 5, 7, 9 Elemental Analysis and e-beam (Straszheim / Kramer)

Week 13 / Nov 12, 14 High pressure generation, gauging and measurements (Bud'ko) Nov 16 Specific heat under pressure (Gati)

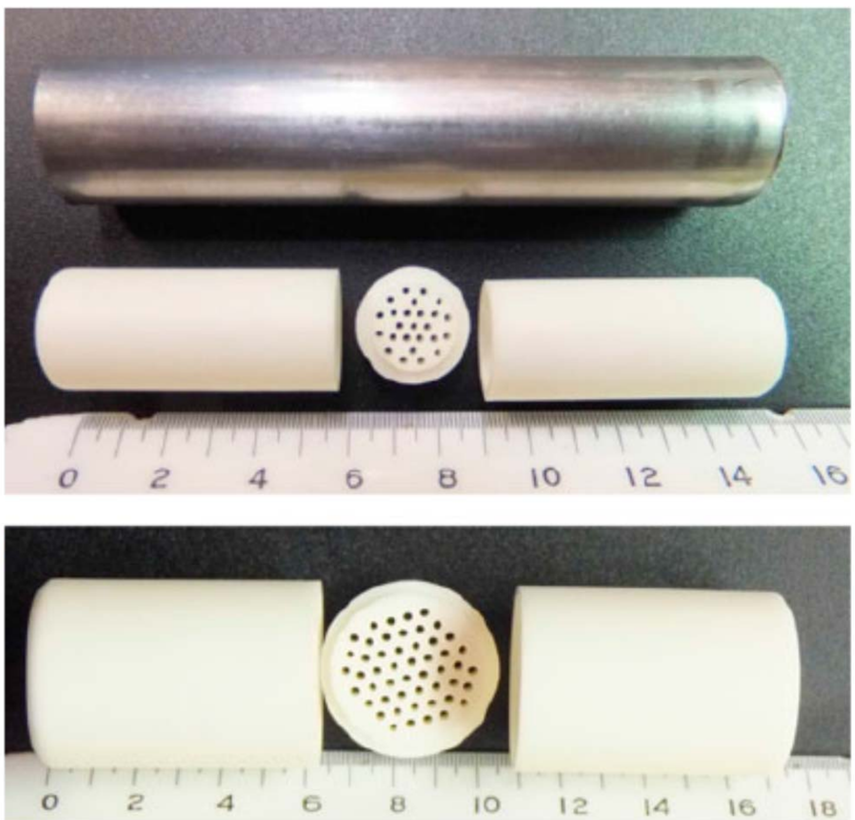
Week 14 / Nov 26, 28, 30 Electrical resistivity measurements (Tanatar)

We were able to work with, and contain, this melt with a combination of Al_2O_3, fritted crucibles that we seal in Ta tubing.

Use of frit-disc crucibles for routine and exploratory solution growth of single crystalline samples

Paul C. Canfield, Tai Kong, Udhara S. Kaluarachchi and Na Hyun Jo

(a)
(b)



PHILOSOPHICAL MAGAZINE, 2016
VOL. 96, NO. 1, 84–92
http://dx.doi.org/10.1080/14786435.2015.1122248

Next Semester 590B Spring 2019

Topics to cover in no particular order

Crystal growth and sample synthesis