

LA-UR-15-21924

Approved for public release; distribution is unlimited.

Title: Macro-Segregation in Uranium-6wt%Niobium

Author(s): Aikin, Robert M. Jr.

Intended for: TMS 2015 Annual Meeting, 2015-03-16 (Orlando, Florida, United States)

Issued: 2015-03-16

Disclaimer:

Los Alamos National Laboratory, an affirmative action/equal opportunity employer, is operated by the Los Alamos National Security, LLC for the National Nuclear Security Administration of the U.S. Department of Energy under contract DE-AC52-06NA25396. By approving this article, the publisher recognizes that the U.S. Government retains nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes. Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy. Los Alamos National Laboratory strongly supports academic freedom and a researcher's right to publish; as an institution, however, the Laboratory does not endorse the viewpoint of a publication or guarantee its technical correctness.

Macro-Segregation in Uranium-6wt%Niobium

Robert M. Aikin Jr.

Presented by John W. Gibbs

Los Alamos National Laboratory
Materials Science & Technology: Metallurgy (MST-6)

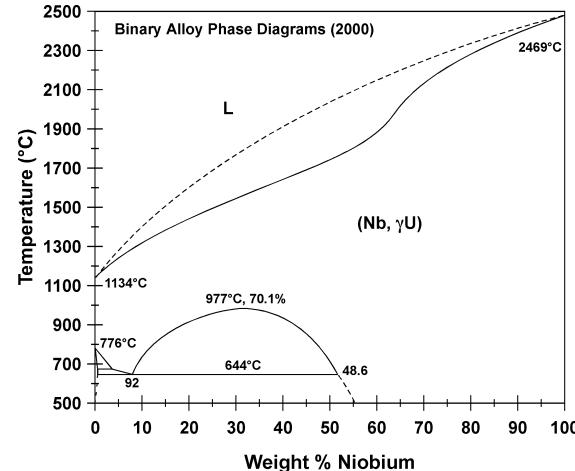
2015 TMS Annual Meeting

March 17, 2015

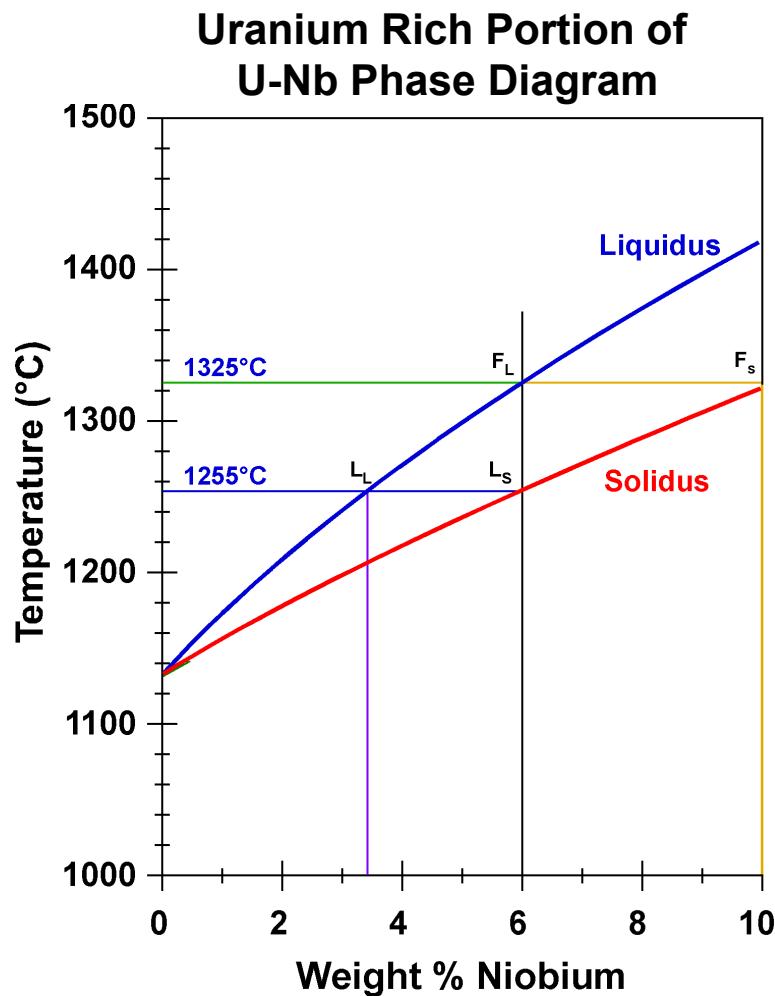


Motivation

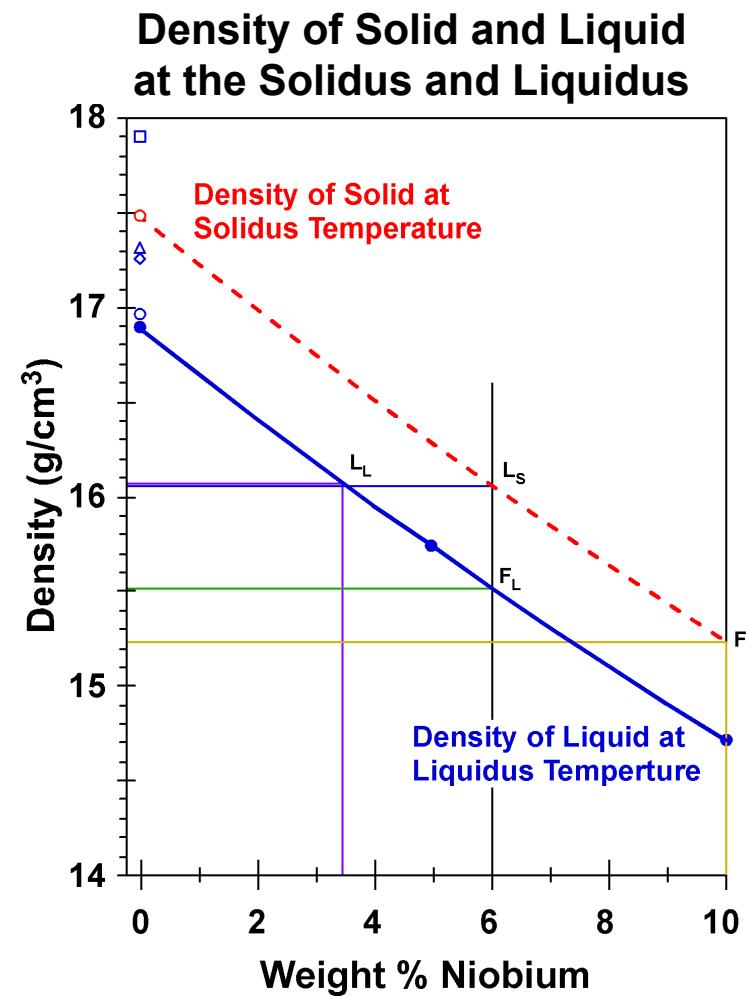
- Historically U-Nb alloys are produced by vacuum arc-remelting (VAR) and subsequent wrought processing, but it would be convenient to produce shape castings by vacuum induction melting (VIM)
- Chemical macro-segregation of the Nb and micro-porosity remains a issue for castings produced by VIM
- This study aims to better understand macro-segregation in cast U-6wt%Nb
 - Influence of solidification time
 - Influence of casting thickness
 - Influence of solidification against and with gravity
- Understanding of solidification behavior is complicated by the fact that allotropic transformations wipe out solidification microstructure



Density Changes During Solidification



Phase diagram after:
B.A. Rogers, et al, Trans. Metall. Soc. AIME (1958)
D. J. Thoma, unpublished data (2004)



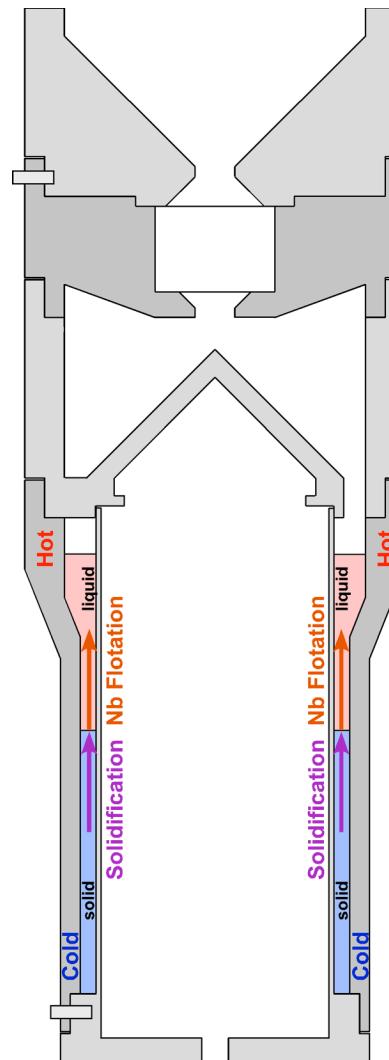
- Liquid density from W.D. Drotning, *High Temp. High Press.*, 14, 253-258 (1982)
- Solid density estimated from published pure U density and Drotning's U-Nb composition dependence of liquid

Growth Direction and Segregation

Assume Macro-Segregation by Buoyancy Driven Flow

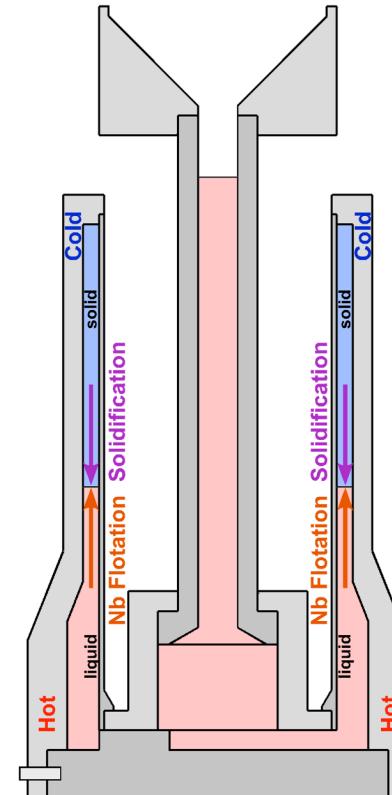
Conventional Mold Design

- Top-to-bottom fill
- Bottom-to-top solidification
- **Solidification front 'chases' Nb flotation**

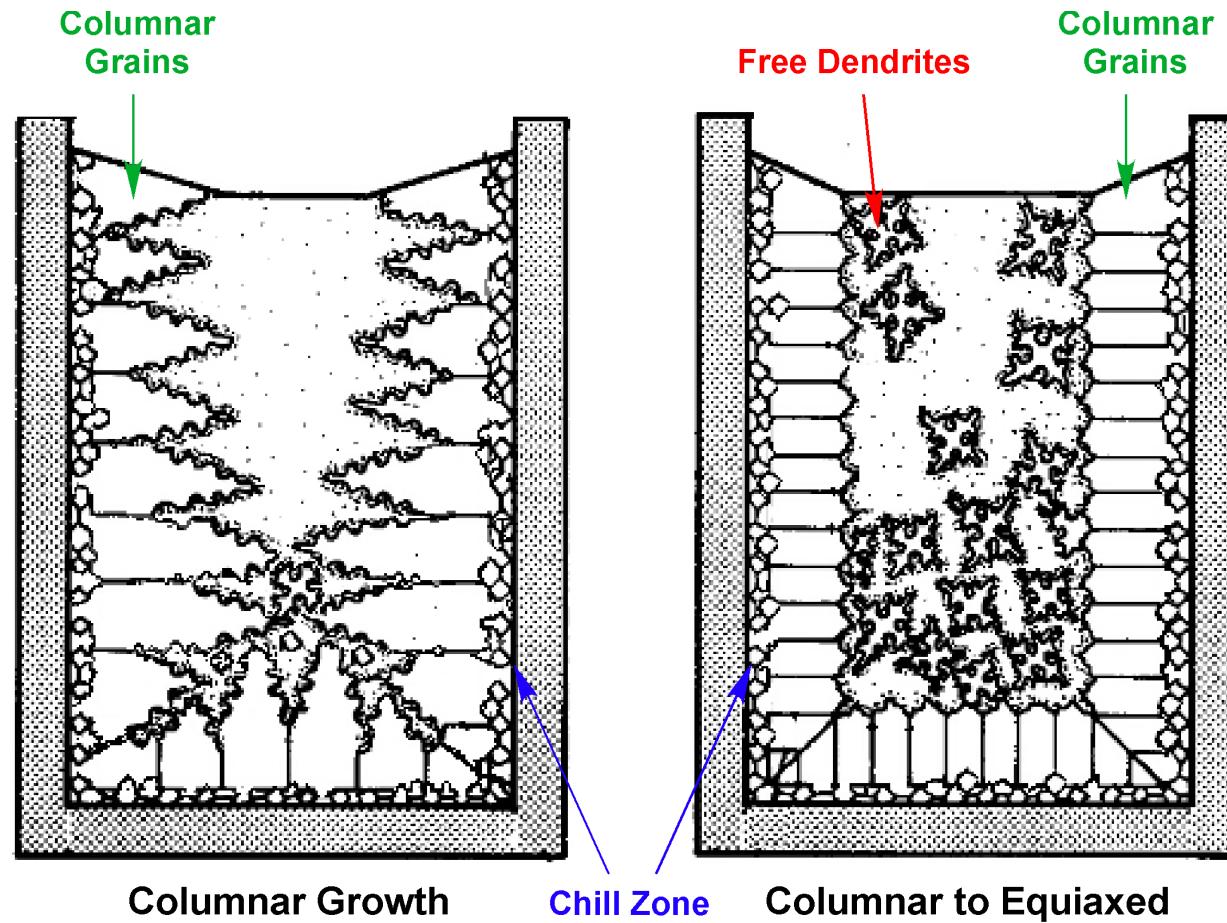


Solidification-Down Mold Design

- Bottom-to-top fill
- Top-to-bottom solidification
- **Solidification direction is opposite Nb flotation**
- Need to keep down-sprue filled and liquid to maintain pressure on casting and feeder



Columnar vs. Free-Dendritic Growth

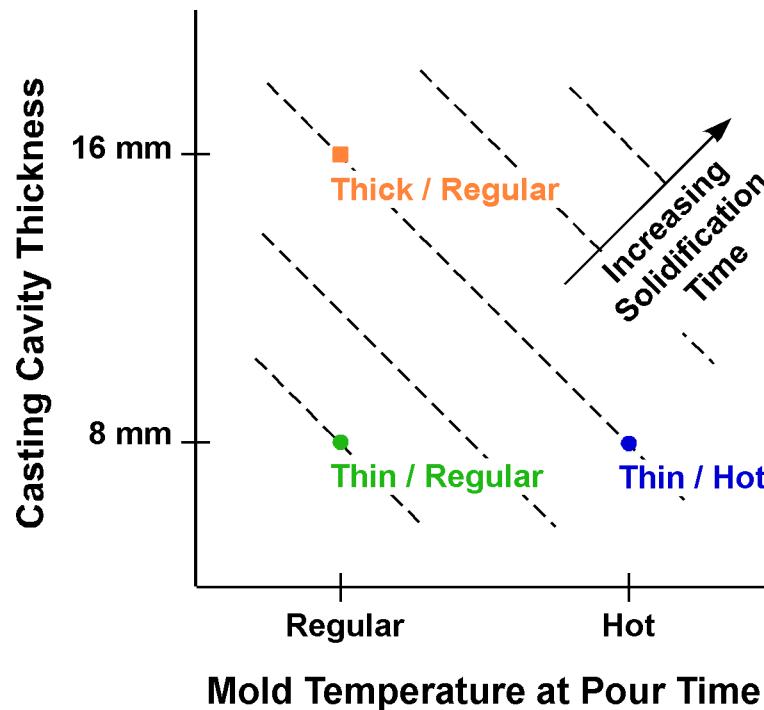


after W. Kurz and D.J. Fisher, Fundamentals of Solidification, Trans Tech Pub (1984).

Experiment Design

Look at three aspects of macro-segregation:

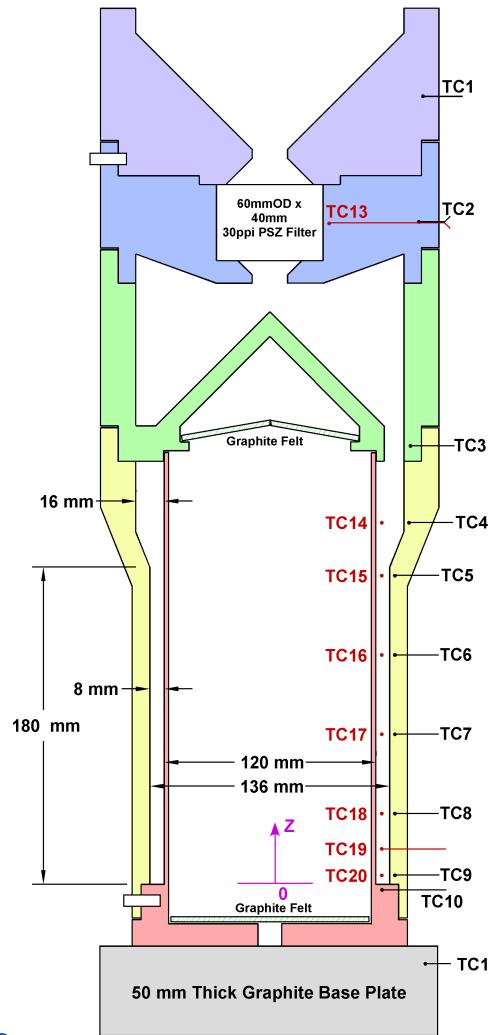
- 1) Influence of solidification time
- 2) Influence of casting thickness
- 3) Solidification-up vs. solidification-down



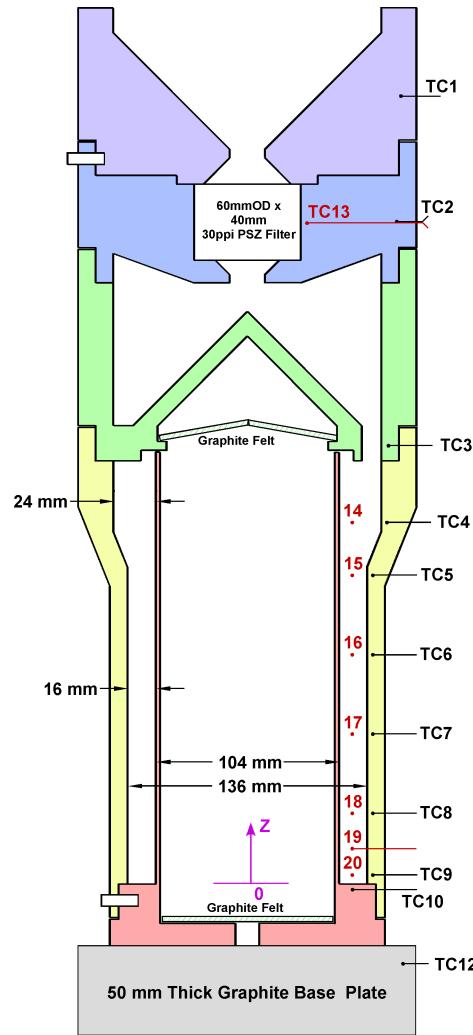
“Hot temperature” was determined by process modeling to try to give same solidification time as the Thin/Regular

Comparison of Molds for Cylindrical Castings

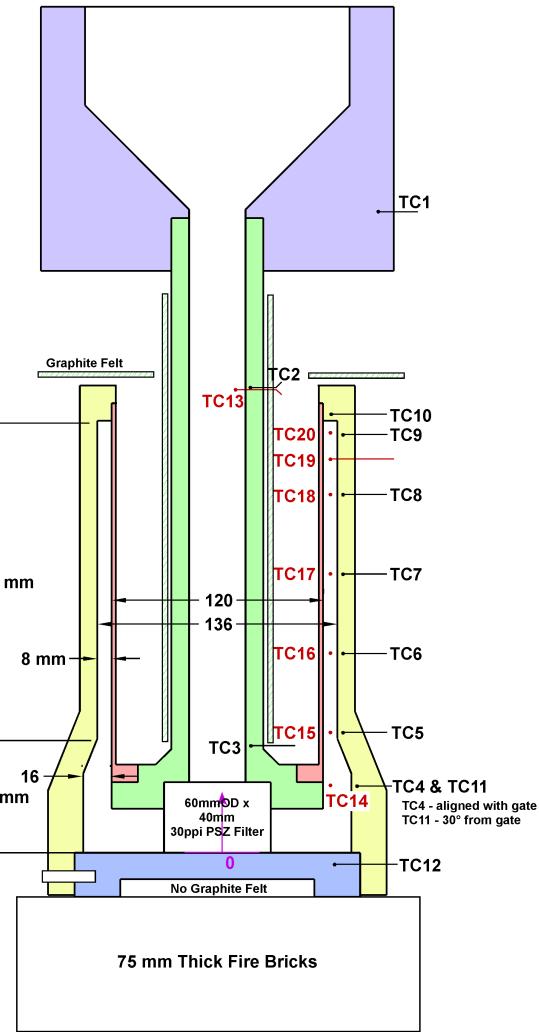
Thin Wall (8 mm)



Thick Wall (16 mm)



Solidification Down (8 mm)



Locations of type-K thermocouples in the mold are indicated in black
 Locations of type-C thermocouples in the casting cavity are indicated in red

Castings Details

- Mold made from fine-grained isotropically pressed graphite (Stackpole 2020)
- Mold coated with Yttria with a cellulose binder (Type-Y from ZYP Coatings)
- Stainless-steel sheathed Type K thermocouples inserted in holes in mold
- Alumina sheathed Type C thermocouples cemented in though-holes in mold wall; bare TC junction in mold cavity
- Wrought U-6Nb plate (scrap) used as charge material
- Same metal pouring temperature used for all 4 castings (1415°C / 60°C superheat)
- Metal filtered though a 30 ppi partially-stabilized zirconia foam filter
- Mold charge and yield

Name	Charge wt (kg)	Casting wt (kg)	Casting Yield
Thick / Regular	28.07	25.28	0.90
Thin / Hot	15.02	13.47	0.90
Thin / Regular	15.13	13.36	0.88
Thin / Top Down	28.01	20.26	0.72

Mold Stack – Solidification Up Mold

Mold stack with thermocouples



Mold stack with 12" diameter induction coils for mold heating and insulation



Bare junction type-C thermocouples in the mold cavity

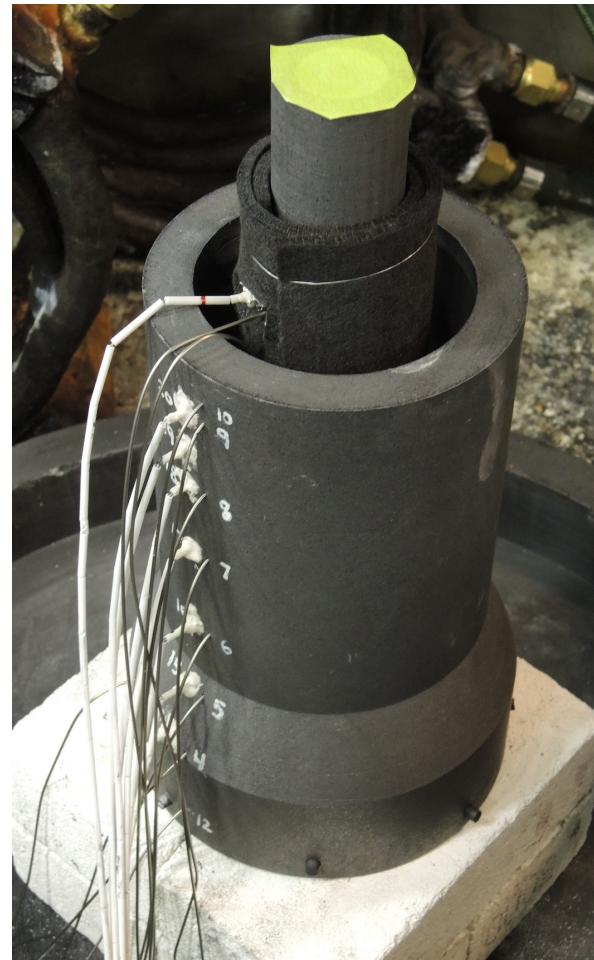


Solidification Down Casting Mold

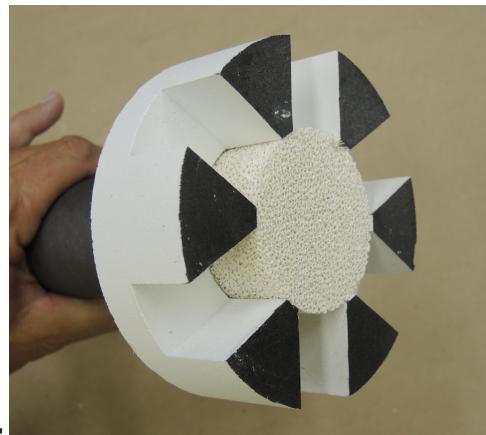
Mold Components Prior to Assembly



Partially Assembled Mold Showing Thermocouples in Mold and in Casting Cavity



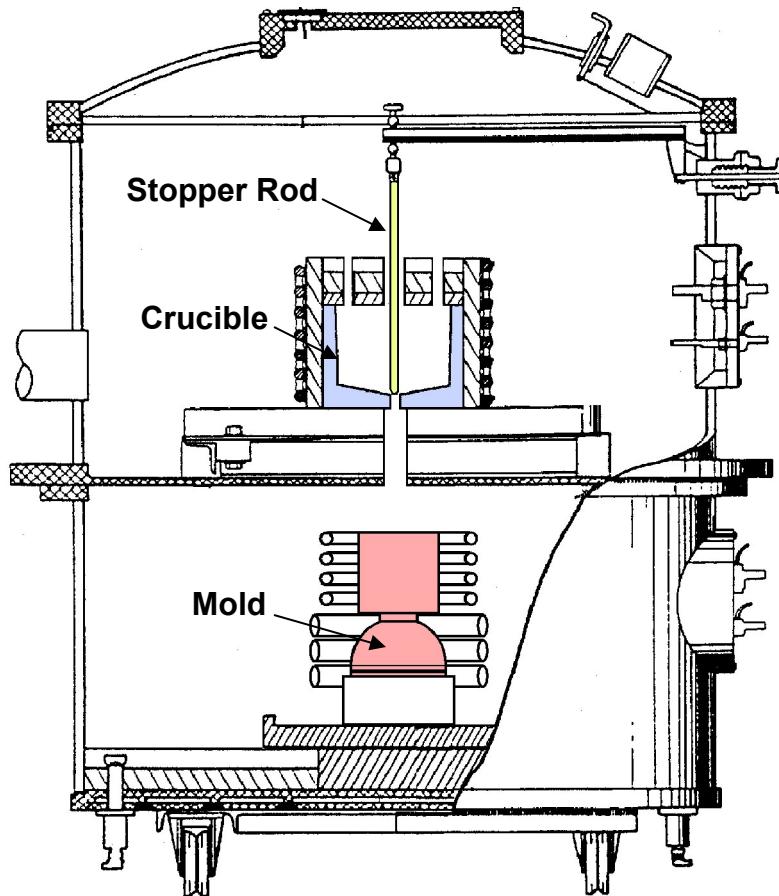
Detail of Sprue/Filter Holder with Porous Zirconia Filter



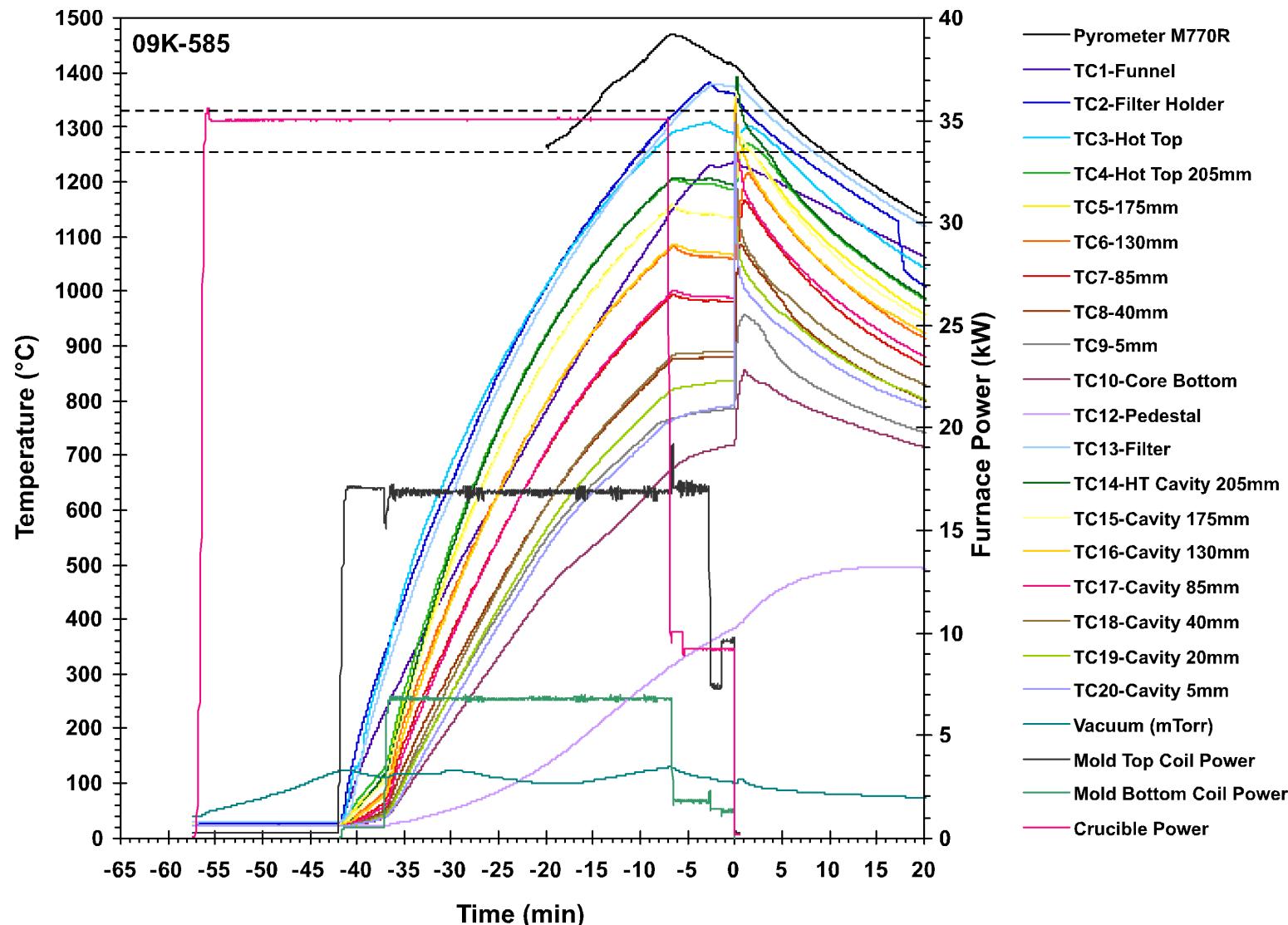
Three Zone Vacuum Induction Furnace

Three separately controlled induction coils:

- Melting crucible coil (35 kW at 9.6 kHz)
- Two mold heating coils (50 kW at 3 kHz)

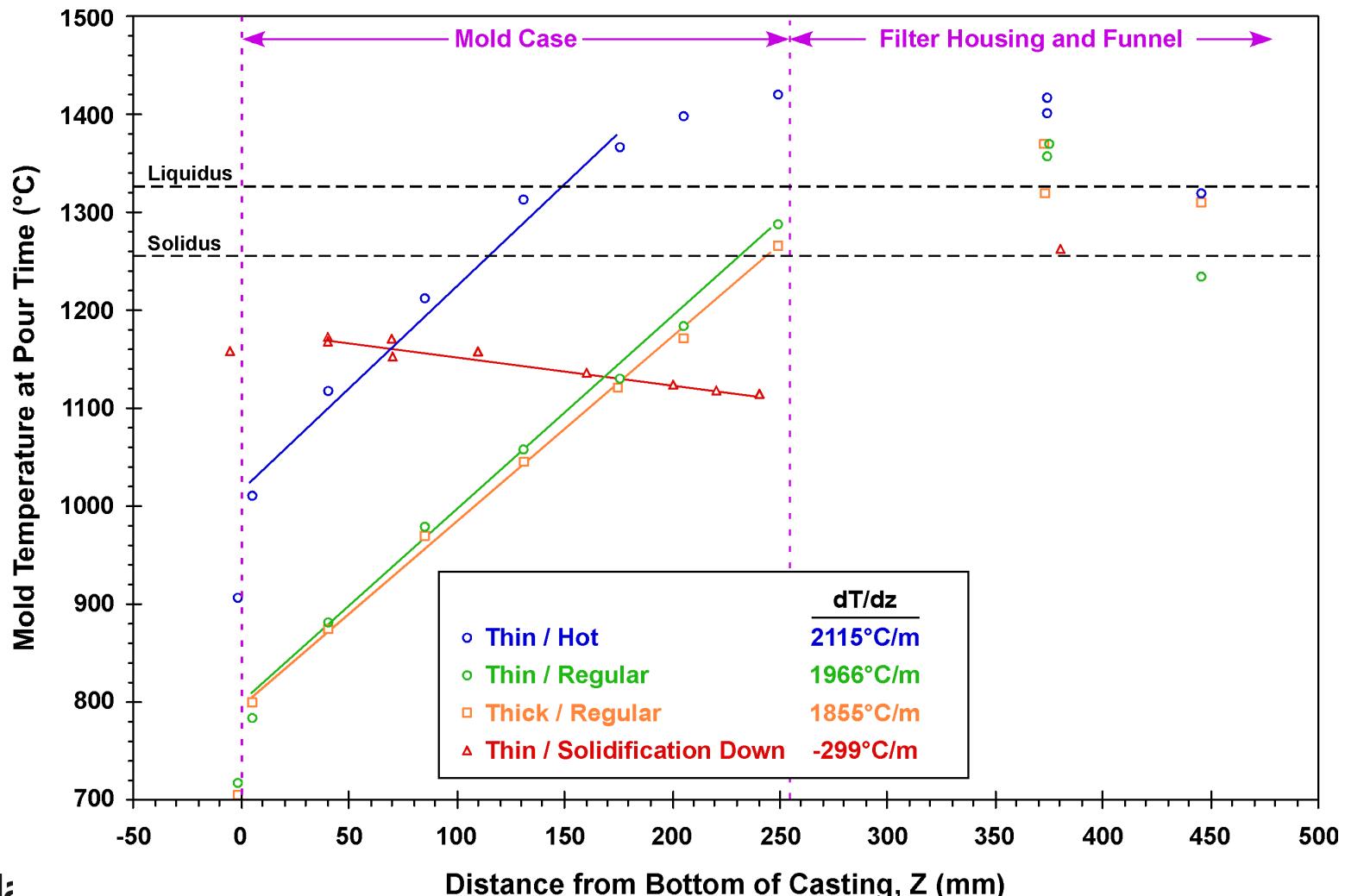


Run Chart - Thin / Regular Mold Temperature



Mold Temperature at Pouring Time

Differing Initial Mold Temperature
Same Metal Pouring Temperature = 1415°C



Cast Parts

**Thick Wall (16 mm)
Casting**



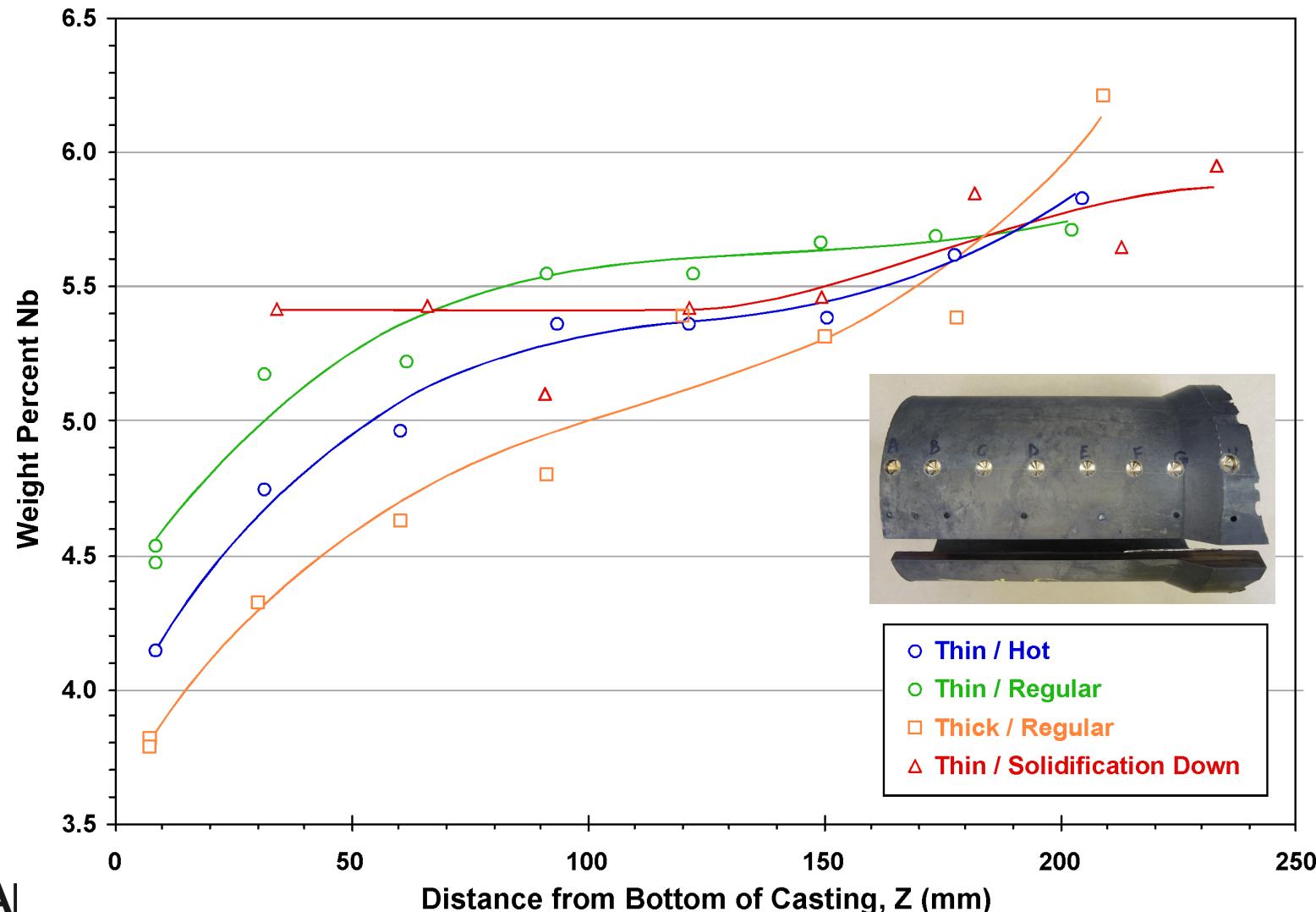
**Solidification Down (8 mm)
Casting**



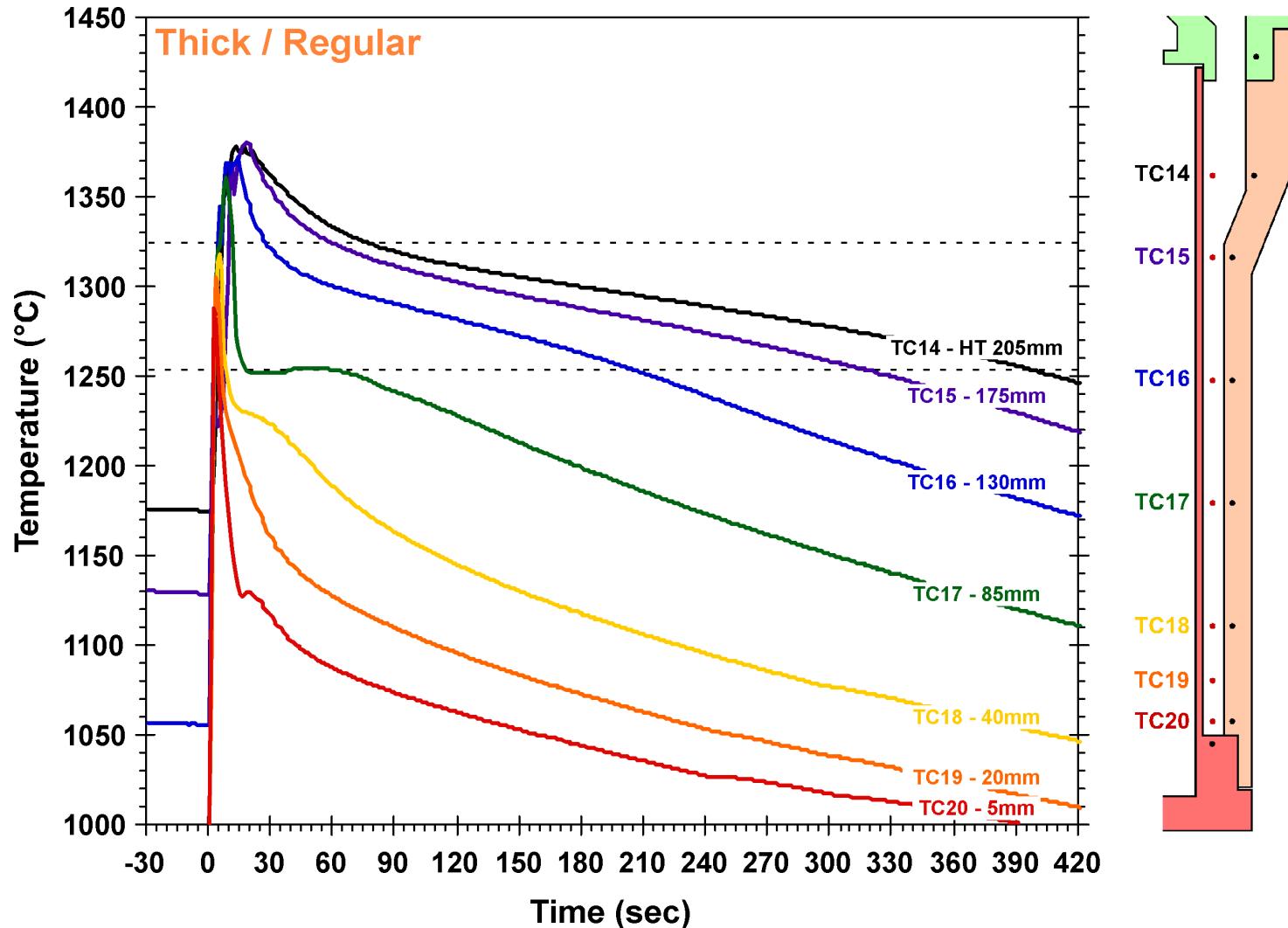
Casting just after removal from mold.
Thermocouple remnants and residual mold
coating can be seen.

Segregation: Thickness vs. Solidification Time

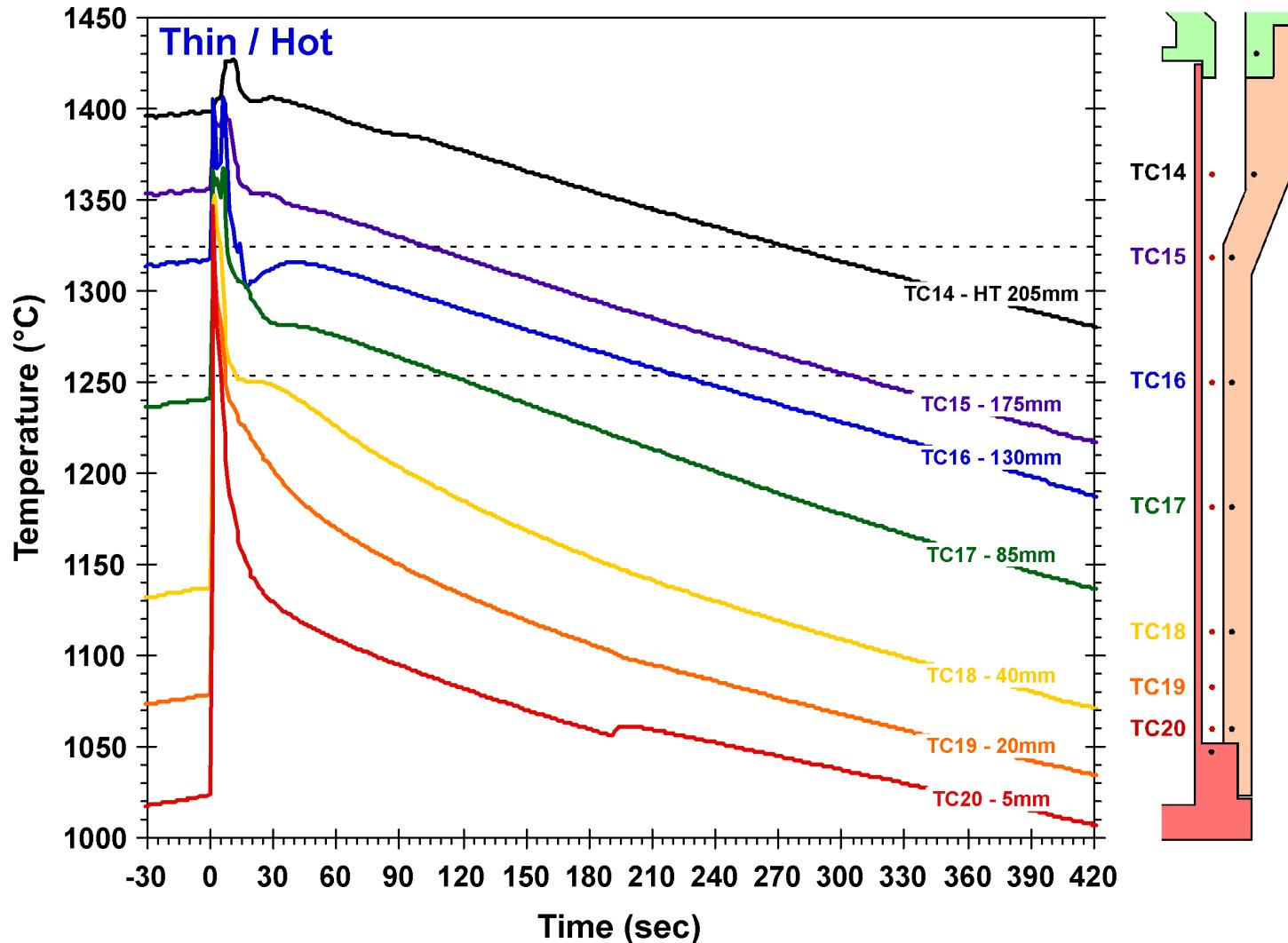
ICP Chemical Analysis Results as Function of Position
Samples from Chips Taken from 9 mm Drill Hole to Casting Center



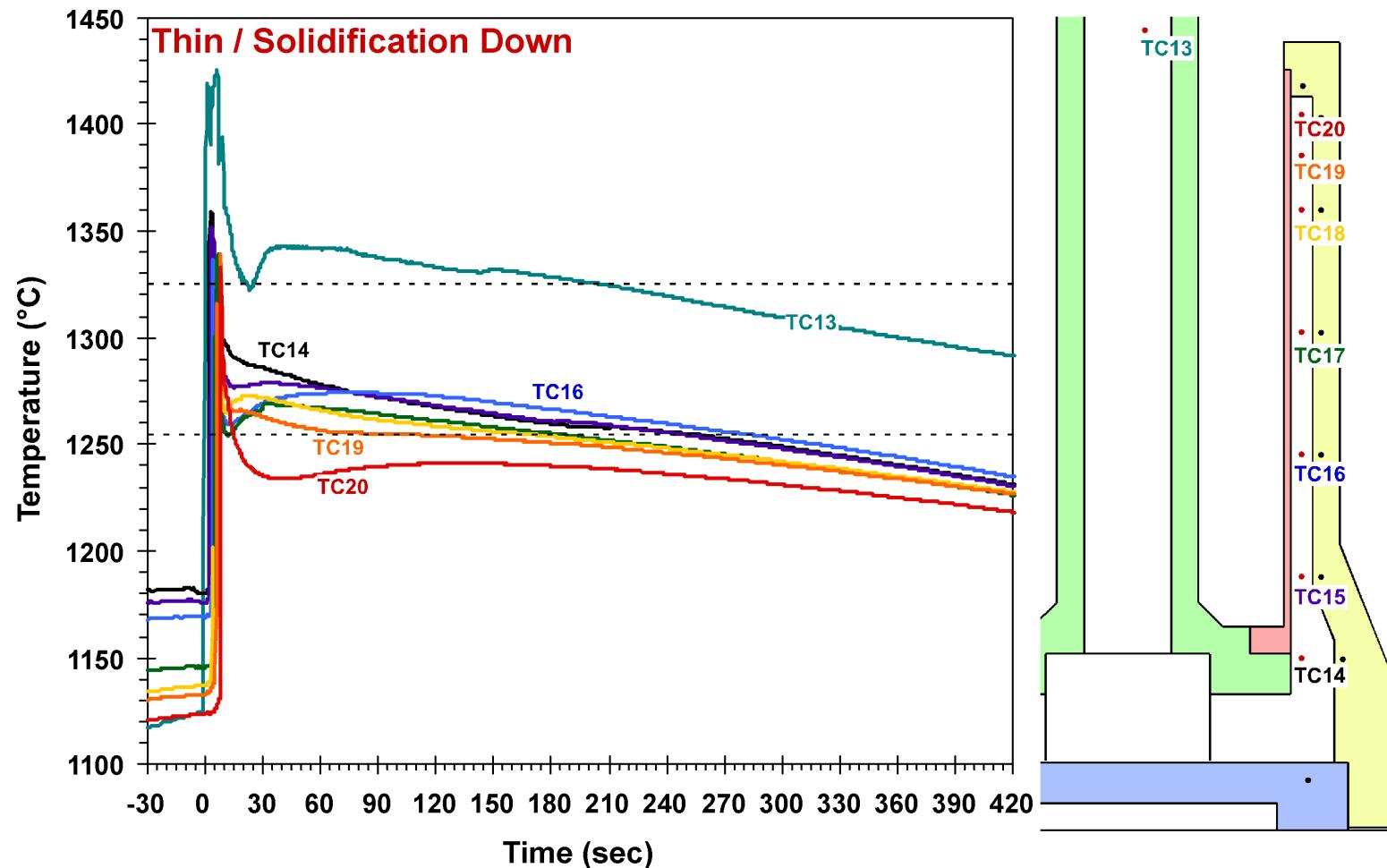
Cooling Curve - Thick / Regular Mold Temperature



Cooling Curve - Thin / Hot Mold Temperature



Cooling Curve - Solidification Down



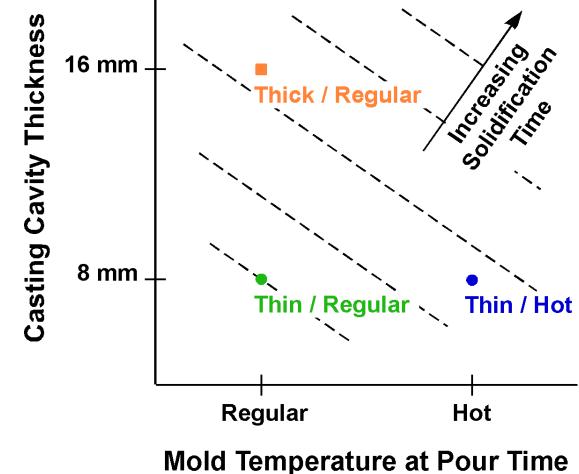
- Mold progressively filled from bottom to top
- Mold *mostly* solidified from top to bottom (TC16 is a hot spot)
- Very shallow thermal gradient; everything mushy at same time

Solidification Time

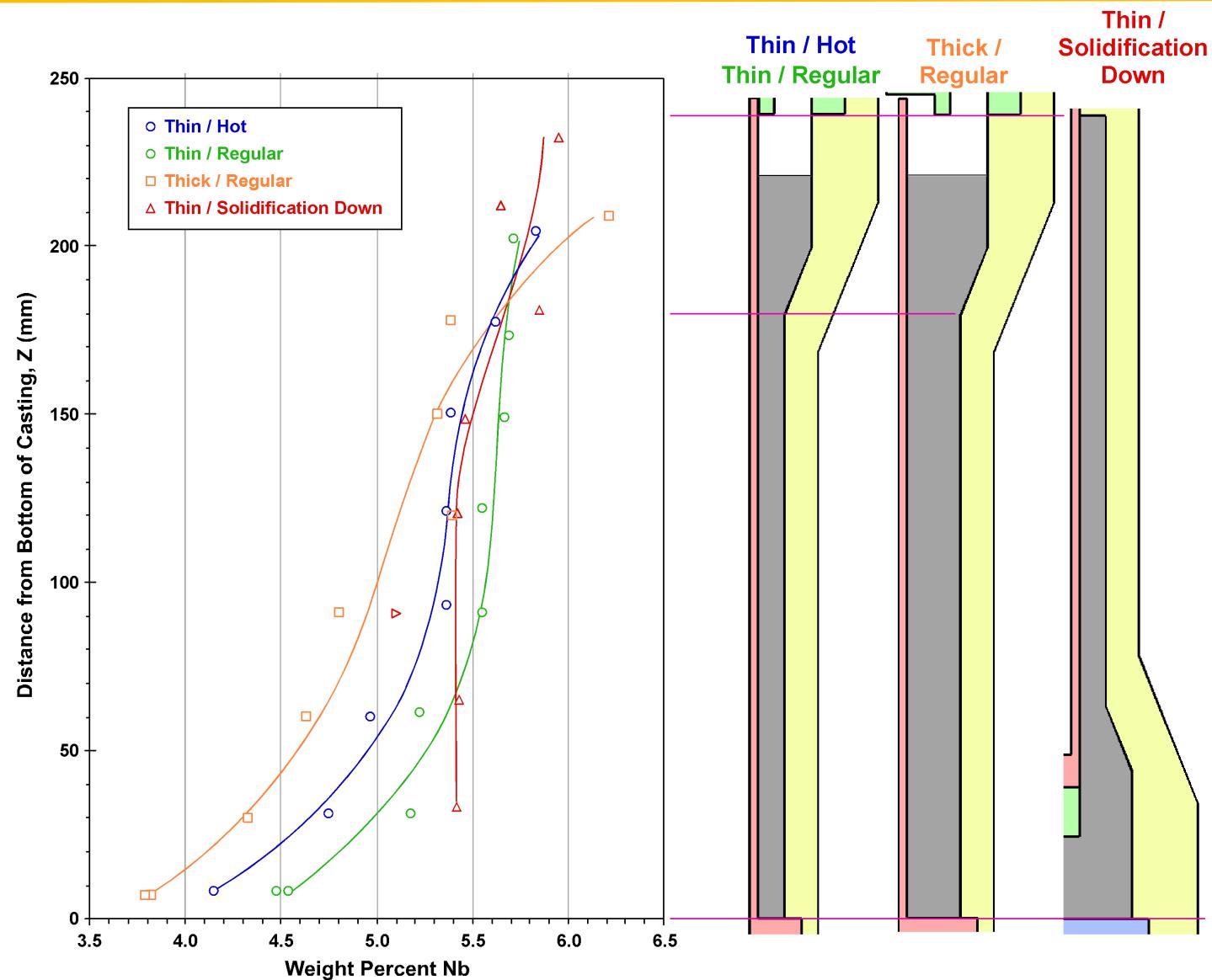
Solidification and Local Solidification Time from Cooling Curves

Solidification Time (s)	Thermocouple ID	Cavity							Down Sprue
		TC20	TC19	TC18	TC17	TC16	TC15	TC14	
		Distance from Bottom, Z (mm)	5	20	40	85	130	175	205
Thin/Hot	TC20	5	7	11	109	217	298	506	
Thin/Regular	TC19	3	4	6	14	54	107	206	
Thick/Regular	TC18	4	6	8	59	198	312	389	
Distance from Top (mm)	TC17	5	20	40	85	130	175	205	-25
Distance from Bottom, Z (mm)	TC16	240	225	205	160	115	70	40	270
Thin / Top Down	TC15	17	74	158	176	275	239	244	365
Thin / Hot	TC14								

Local Solidification Time (s)	Thermocouple ID	Cavity							Down Sprue
		TC20	TC19	TC18	TC17	TC16	TC15	TC14	
		Distance from Bottom, Z (mm)	5	20	40	85	130	175	205
Thin/Hot	TC20				7	100	206	211	252
Thin/Regular	TC19				3	9	49	103	165
Thick/Regular	TC18					53	174	264	326
Distance from Top (mm)	TC17	5	20	40	85	130	175	205	-25
Distance from Bottom, Z (mm)	TC16	240	225	205	160	115	70	40	270
Thin / Top Down	TC15	8	69	158	168	269	233	239	636
Thin / Hot	TC14								



Segregation: Thickness vs. Solidification Time



Conclusions

- For U-6wt%Nb castings solidified bottom-to-top:
 - Segregation is most strongly related to solidification time
 - Macro-segregation is most severe in the bottom of the conventional castings; this is the region which also solidifies most rapidly
 - For similar solidification times there does appear to be a thickness effect (for the 8 to 16 mm thickness examined)
- For casting solidified down (top-to-bottom):
 - Macro-segregation is mostly eliminated
 - A higher thermal gradient than obtained in this experiment would be beneficial to better cast structure and a better cast part
- The observed macro-segregation is consistent with free-dendritic growth with flotation (buoyancy driven and/or filling driven advection of liquid?)