

# “Grain Refinement of Al-Si Alloys by Nb-B Inoculation. Part 1: Concept Development and Effect on Binary Alloys. Part 2: Application to Commercial Alloys”

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**UK**



# Grain refinement of Al-Si alloys by Nb-B inoculation

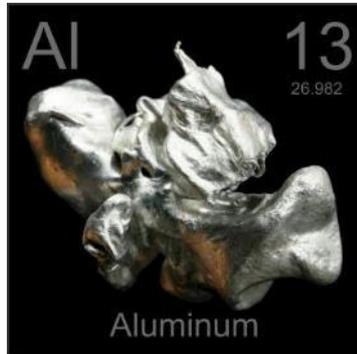
M. Nowak  
L. Bolzoni  
N. Hari Babu

Brunel Centre for Advanced Solidification Technology  
Brunel University London, UK

# Outline

- Grain refinement in Al alloys with Al-5Ti-B
- Concept development
- Application to Al-Si cast alloys
- Al-Nb-B master alloy
- Comparative study between Al-Nb-B and Al-5Ti-B master alloys

# ALUMINIUM ALLOYS



## PROPERTIES

- LOW DENSITY, 2.7 g/cc
- GOOD MECHANICAL PROPERTIES
- HIGH CORROSION RESISTANCE
- HIGH THERMAL CONDUCTIVITY
- LOW ELECTRICAL RESISTIVITY

ALLOYS

WROUGHT

CAST (Al-Si)

GRAIN  
REFINEMENT

IMPROVEMENT

FLUIDITY/CASTABILITY

MECHANICAL PROPERTIES

MACHINABILITY

SURFACE QUALITY

CHEMICAL HOMOGENEITY

REDUCED SHRINKAGE POROSITY

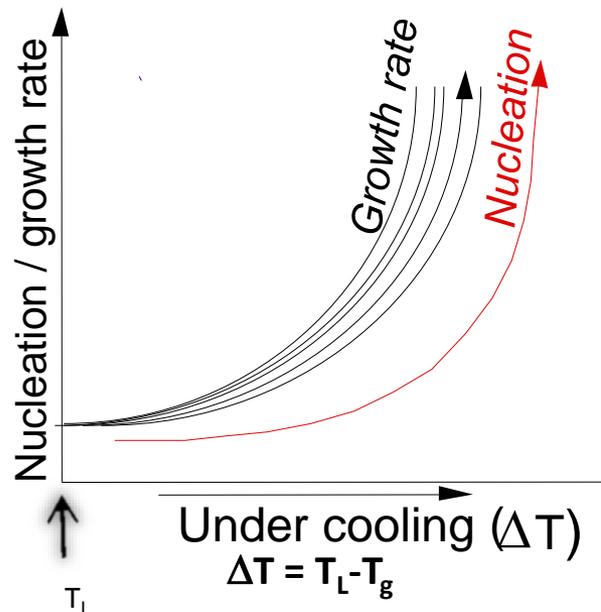
# Factors determining grain size in as cast microstructure

## Nucleation & Growth

Heterogeneous nucleation  
Homogeneous nucleation

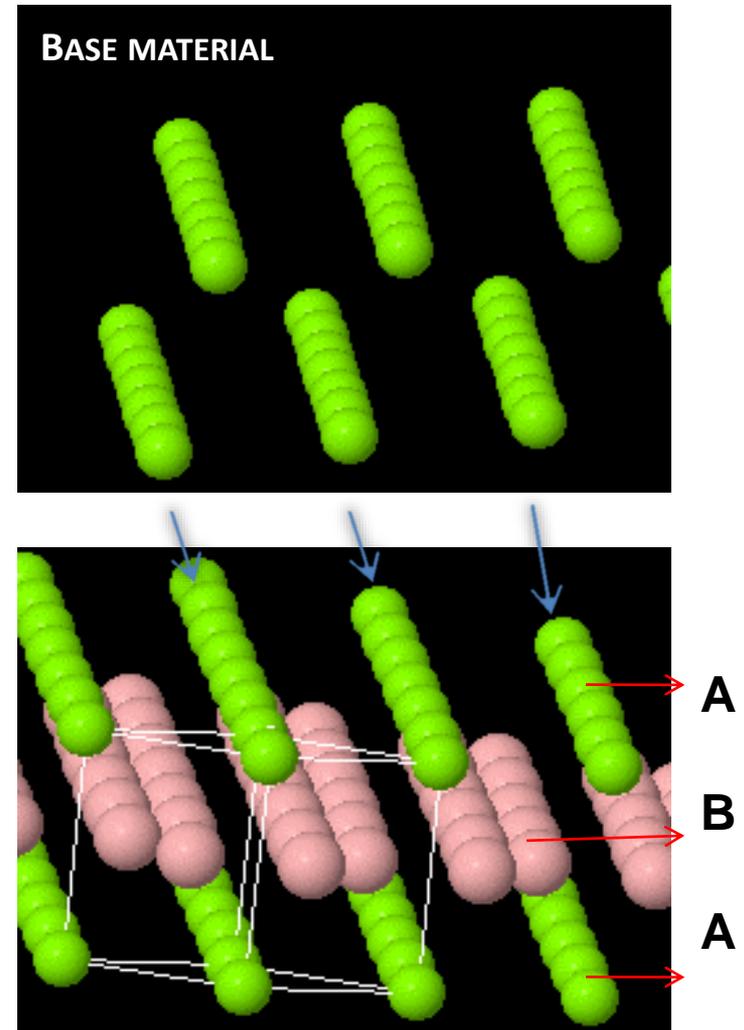
## Growth Kinetics

- Temperature
- Atmosphere/Pressure
- Growth restriction
- Fragmentation
- Cooling rate



# EFFICIENT HETEROGENEOUS NUCLEATION SITES

1. High melting Temp
2. Low lattice mismatch  
(atom position matching)
3. Chemical stability  
(should not react with alloying  
elements)



*N. Hari Babu et al., Nature Materials 2005;4:476*

# GRAIN REFINERS IN ALUMINIUM INDUSTRY

**GRAIN REFINEMENT:** Al-Ti-B

Al-Ti-C

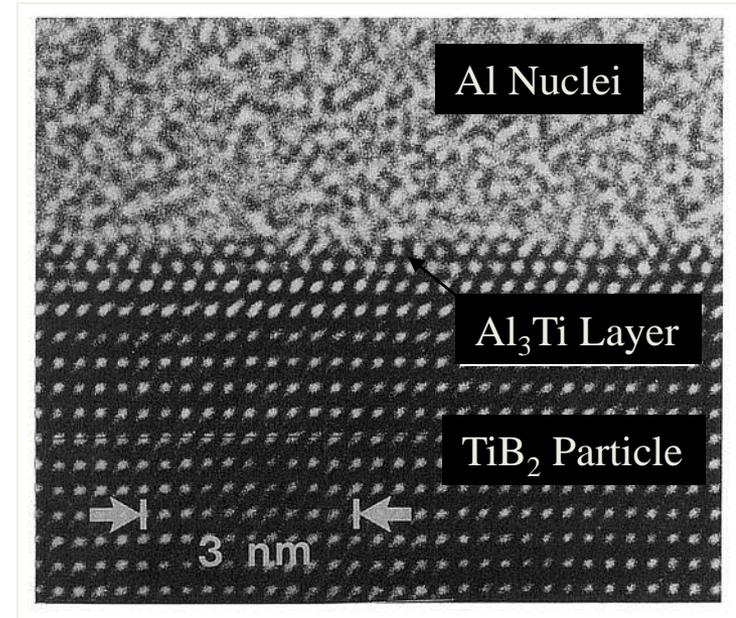
TiB<sub>2</sub> & Al<sub>3</sub>Ti

- Orientation Relationships

$\{111\}Al//\{112\}Al_3Ti//\{001\}TiB_2$

$\langle 110 \rangle Al//\langle 201 \rangle Al_3Ti$

$\langle 110 \rangle Al_3Ti//\langle 110 \rangle TiB_2$



HREM image of Al/Al<sub>3</sub>Ti/TiB<sub>2</sub> interface

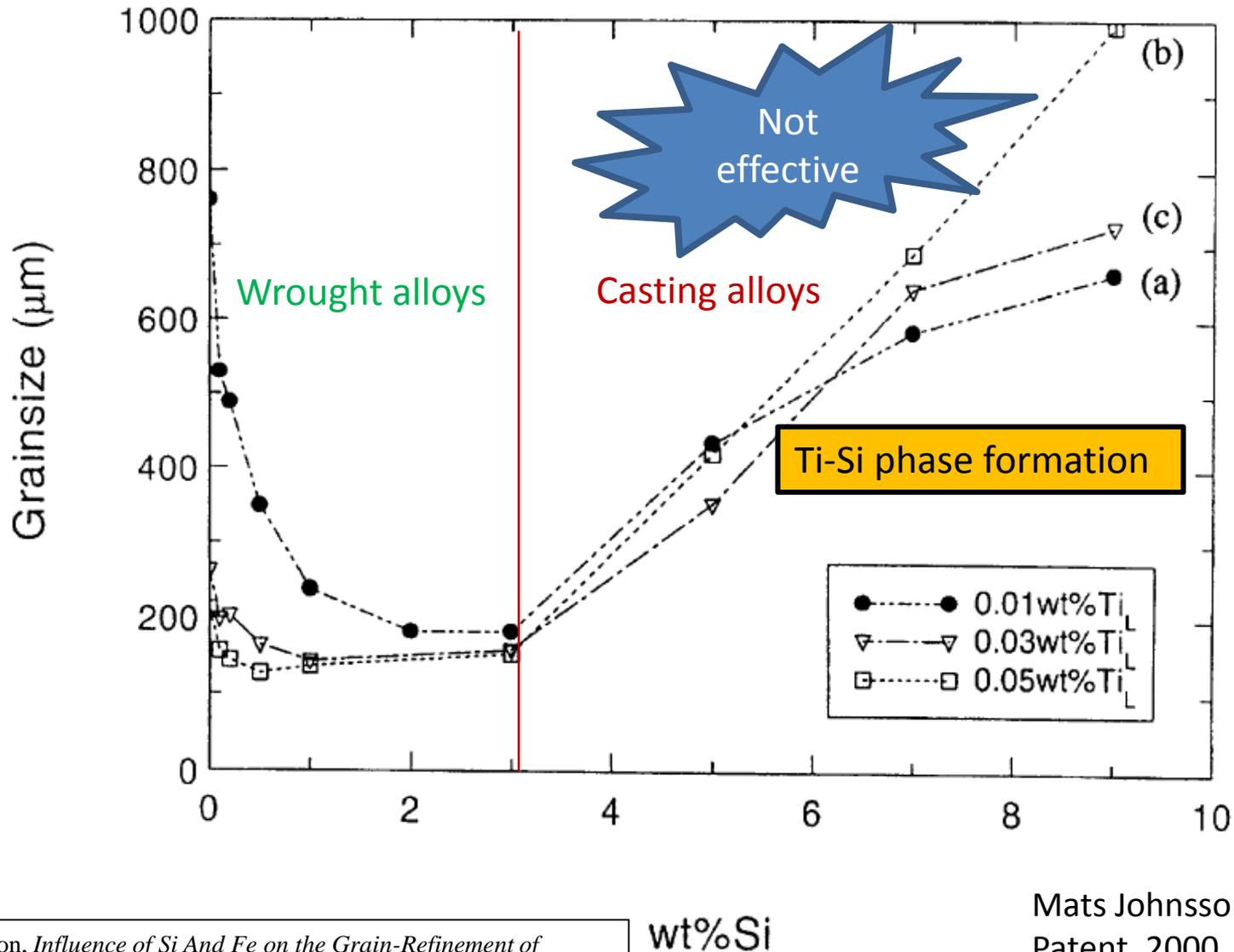
Source: B. J McKay

**MODIFICATION:**

Sr modification of the Si morphology

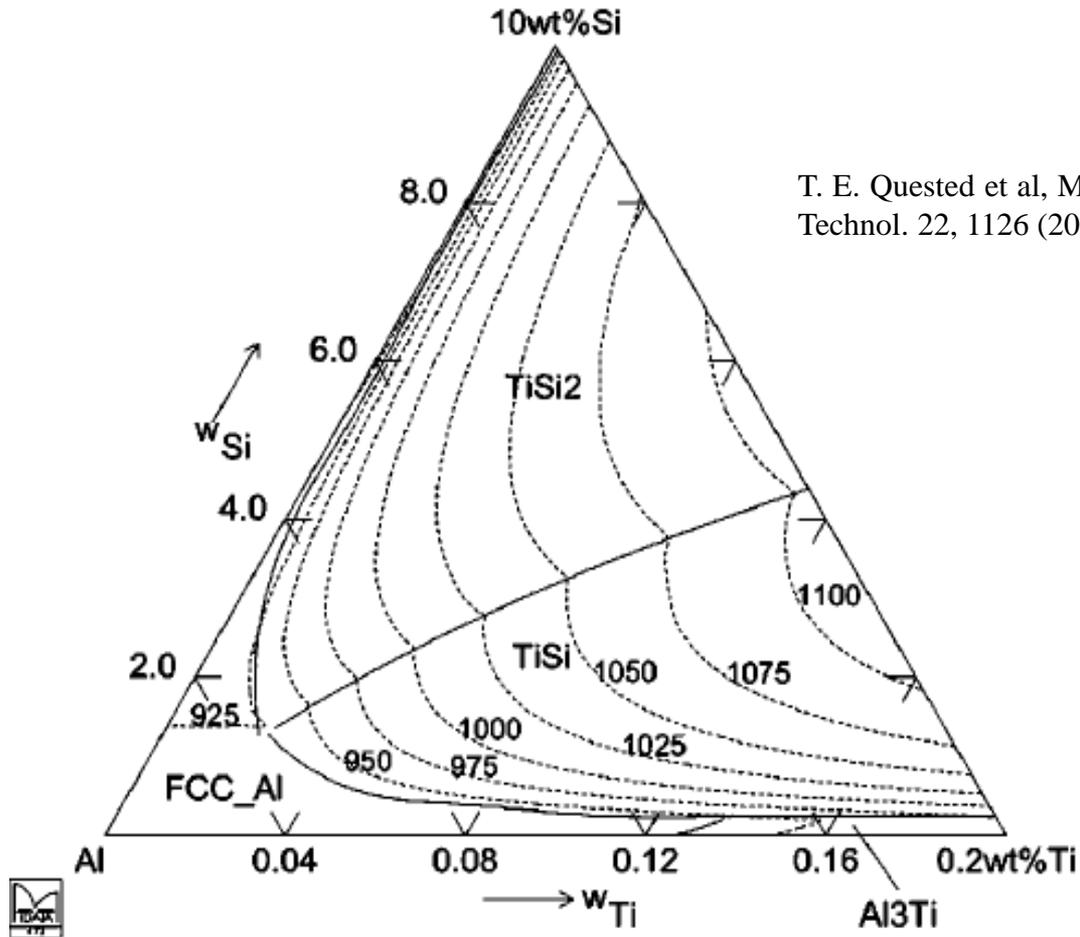
P to nucleate the primary Si particles

# Influence of Al-Ti-B grain refiner for Al-Si alloys



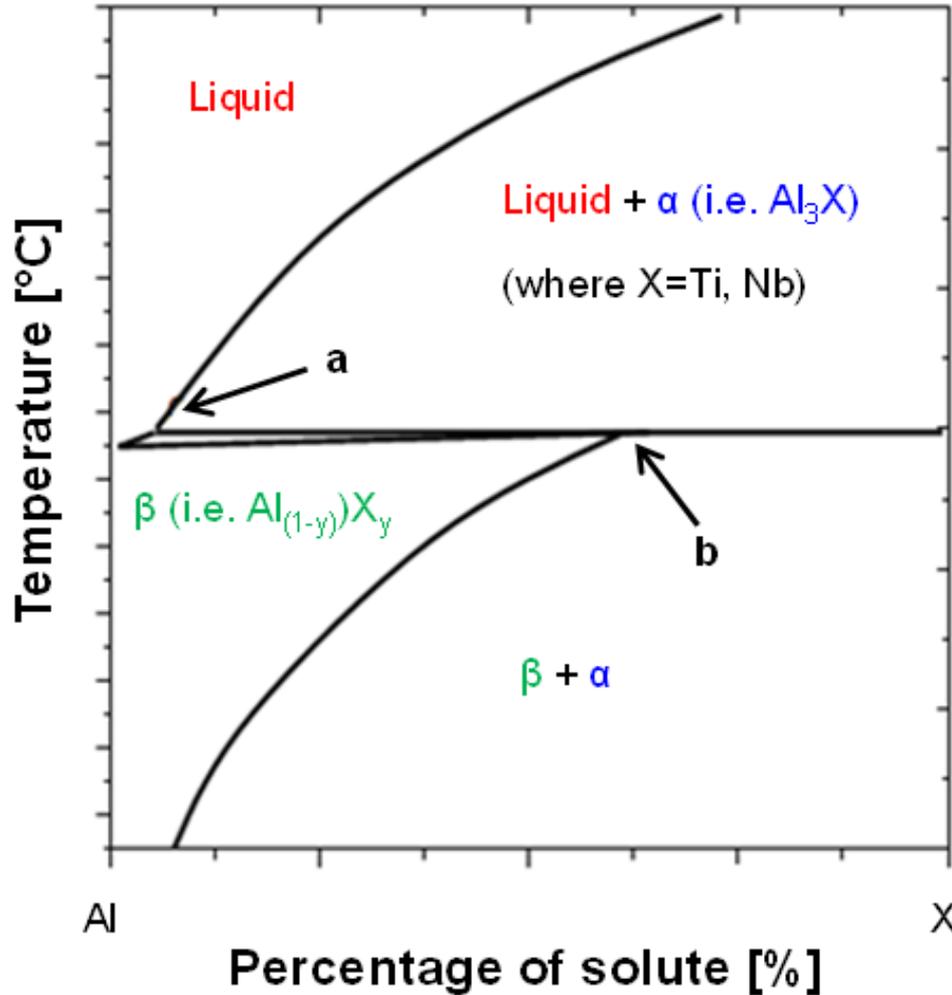
Mats Johansson  
Patent, 2000

# Ti reaction with Si in Al-Si alloys

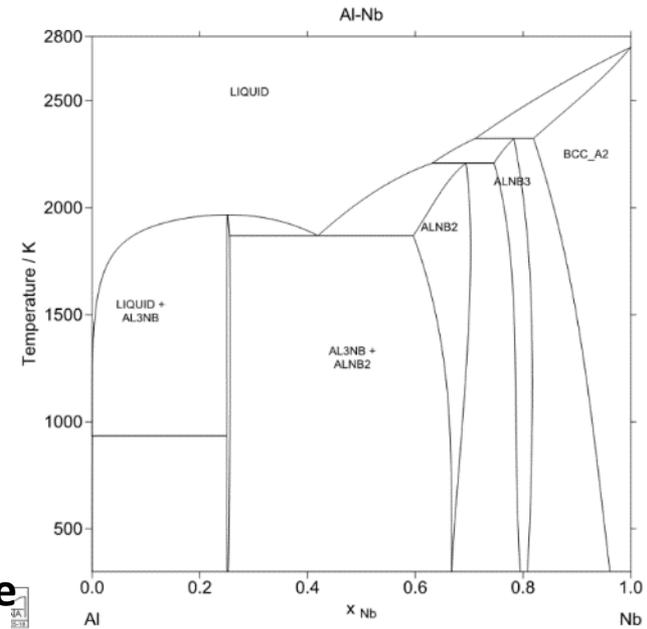
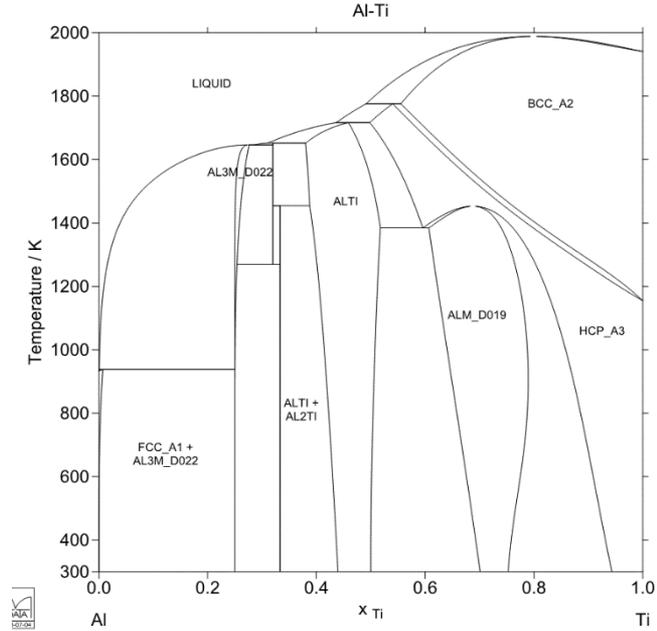


Ti is consumed by the formation of  $\text{TiSi}_2$  and  $\text{TiSi}$

# Analogy between Al-Ti & Al-Nb phase diagrams

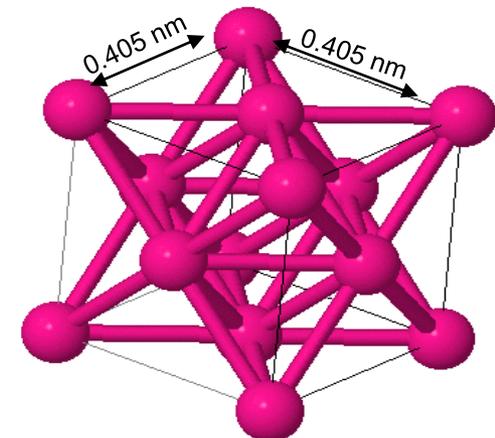
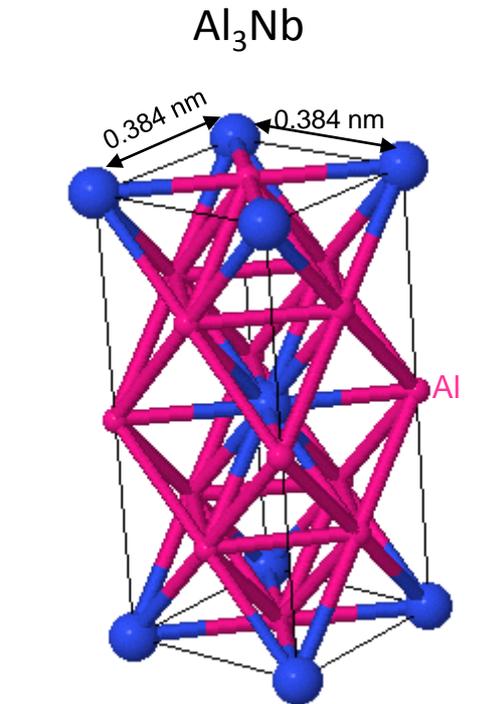


$\text{Al}_3\text{Ti}$   
 $\text{Al}_3\text{Nb}$



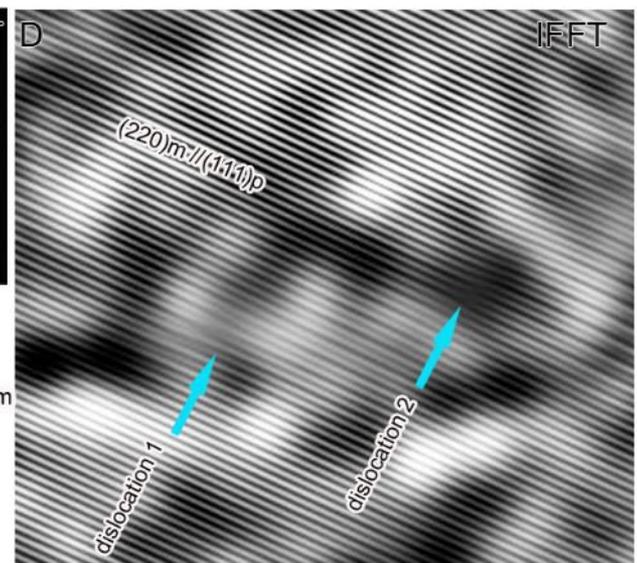
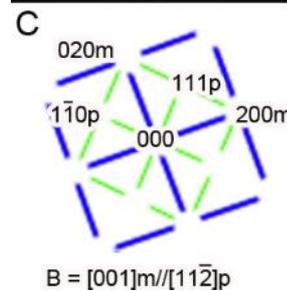
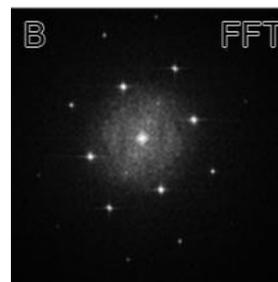
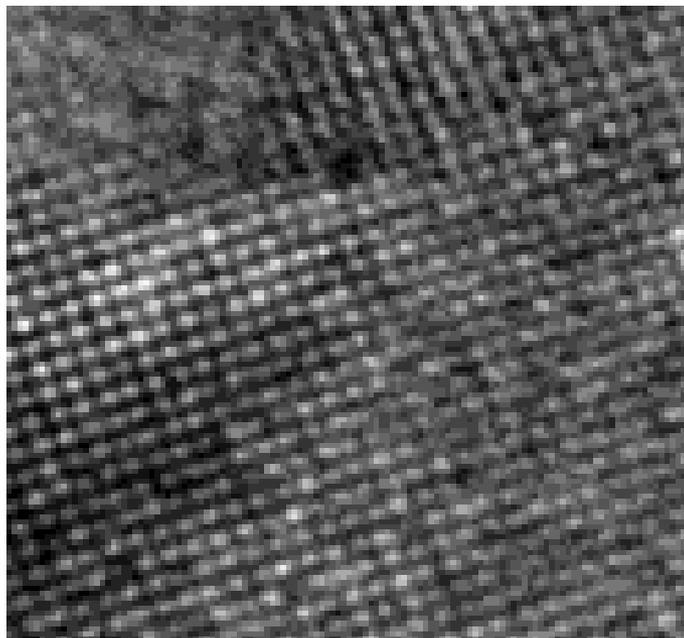
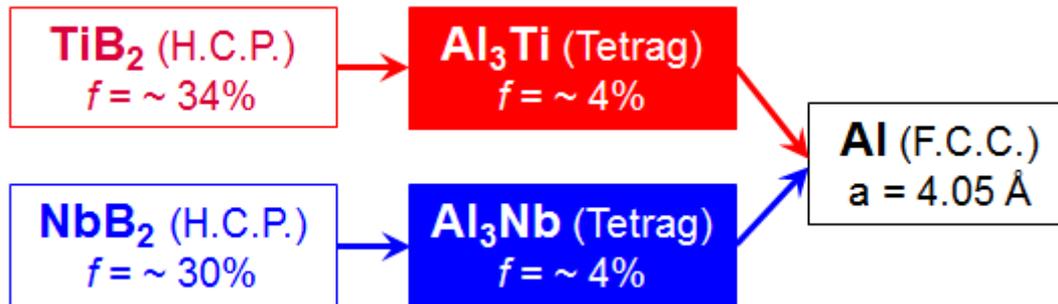
# LATTICE MISMATCH

Element	Phase	Melting Point [°C]	Density [g/cm <sup>3</sup> ]	Lattice structure	Lattice parameter
Aluminium	Al	660	2.70	Face-Centred Cubic	a = 4.050 Å
Titanium	Ti	1668	4.51	Hexagonal	a = 2.950 Å, c = 4.683 Å
	Al <sub>3</sub> Ti	1350	3.36	Tetragonal	a = 3.848 Å, c = 8.596 Å
	TiB <sub>2</sub>	3230	4.52	Hexagonal	a = 3.023 Å, c = 3.220 Å
	TiC	3160	4.93	Face-Centred Cubic	a = 4.330 Å
Niobium	Nb	2468	8.57	Body-Centred Cubic	a = 3.300 Å
	Al <sub>3</sub> Nb	1680	4.54	Tetragonal	a = 3.848 Å, c = 8.615 Å
	NbB <sub>2</sub>	3036	6.98	Hexagonal	a = 3.102 Å, c = 3.285 Å
	NbC	3490	7.82	Face-Centred Cubic	a = 4.430 Å



Al (face centred cubic)

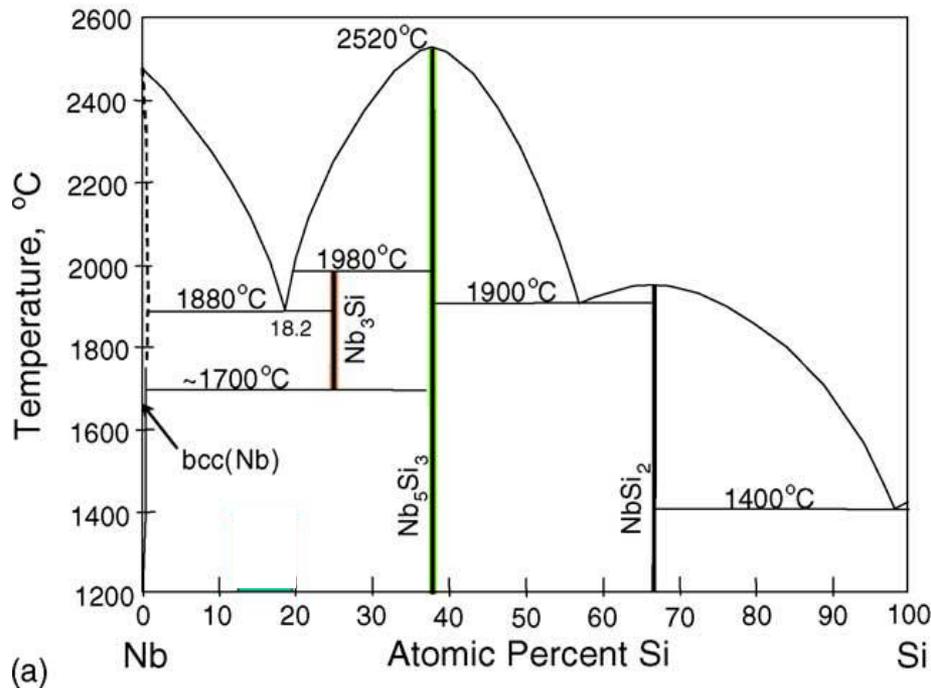
# Low Lattice Mismatch – Coherent Interface



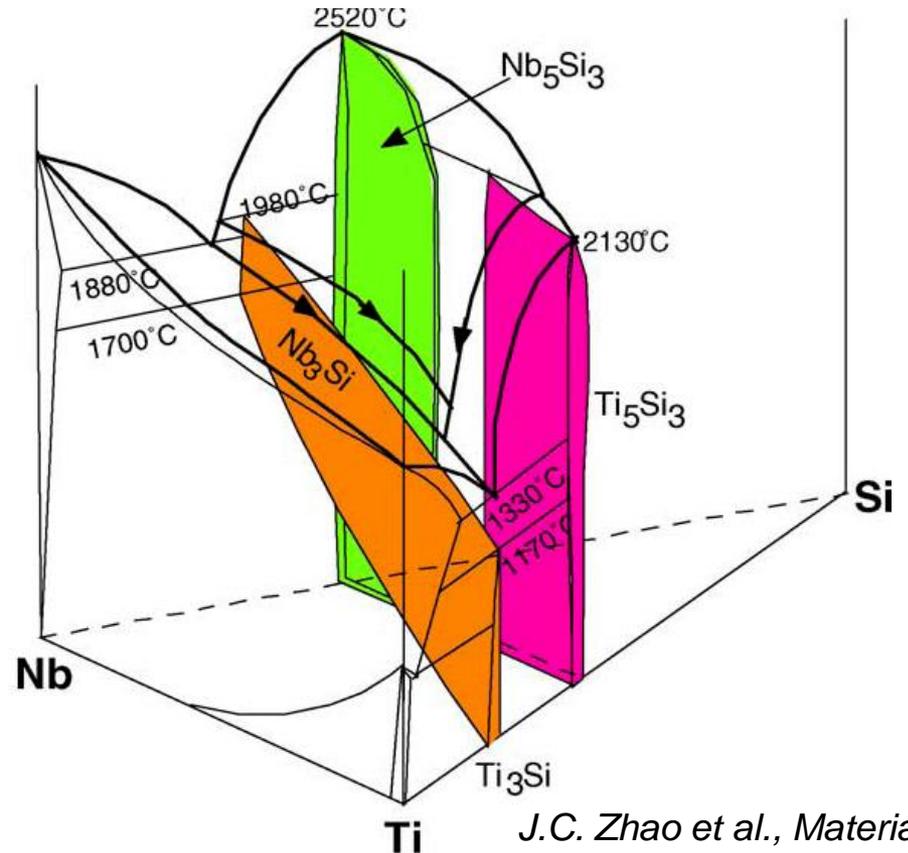
**Lattice mismatch  $\sim 0.9\%$**

# Nb chemical stability with Si

## Nb–Si binary phase diagram



## Nb–Ti–Si ternary system



*J.C. Zhao et al., Materials Science and Engineering A 2004;372:21*

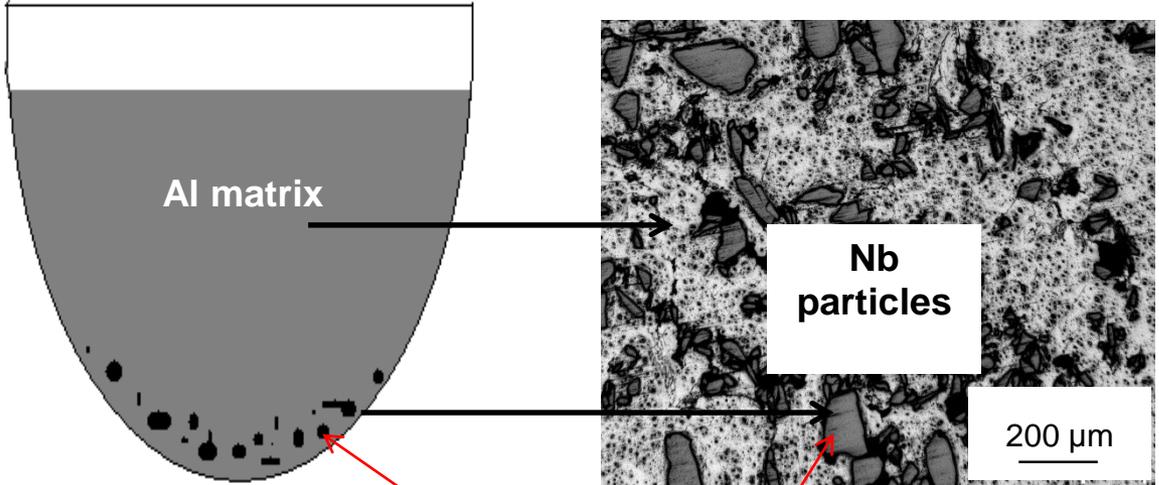
*J.L. Murray and A. J. Alister, Bulletin of Alloy Phase Diagrams 1984;5:74*

**Nb silicides form at higher temperature than Ti silicides thus preventing poisoning**

# Addition of Nb metal powder to liquid Al

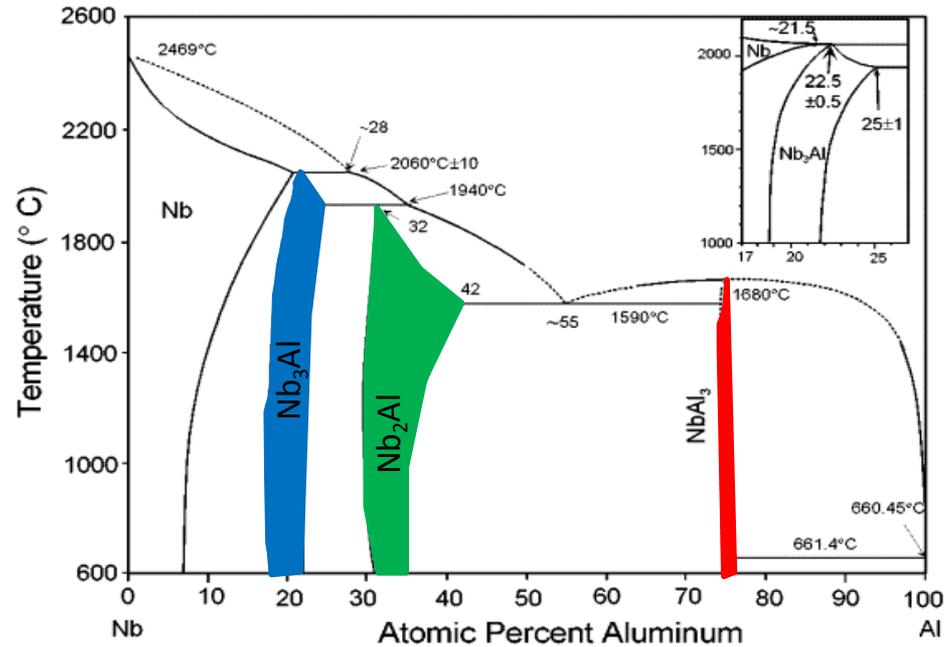
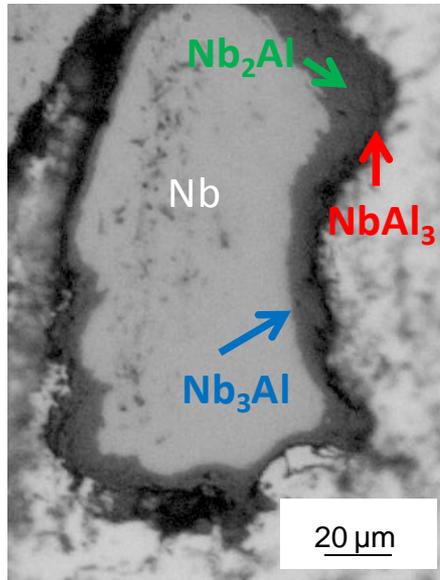


750 - 800 C



Unreacted Nb metallic particulates

# Poor dissolution of Nb in liquid Al

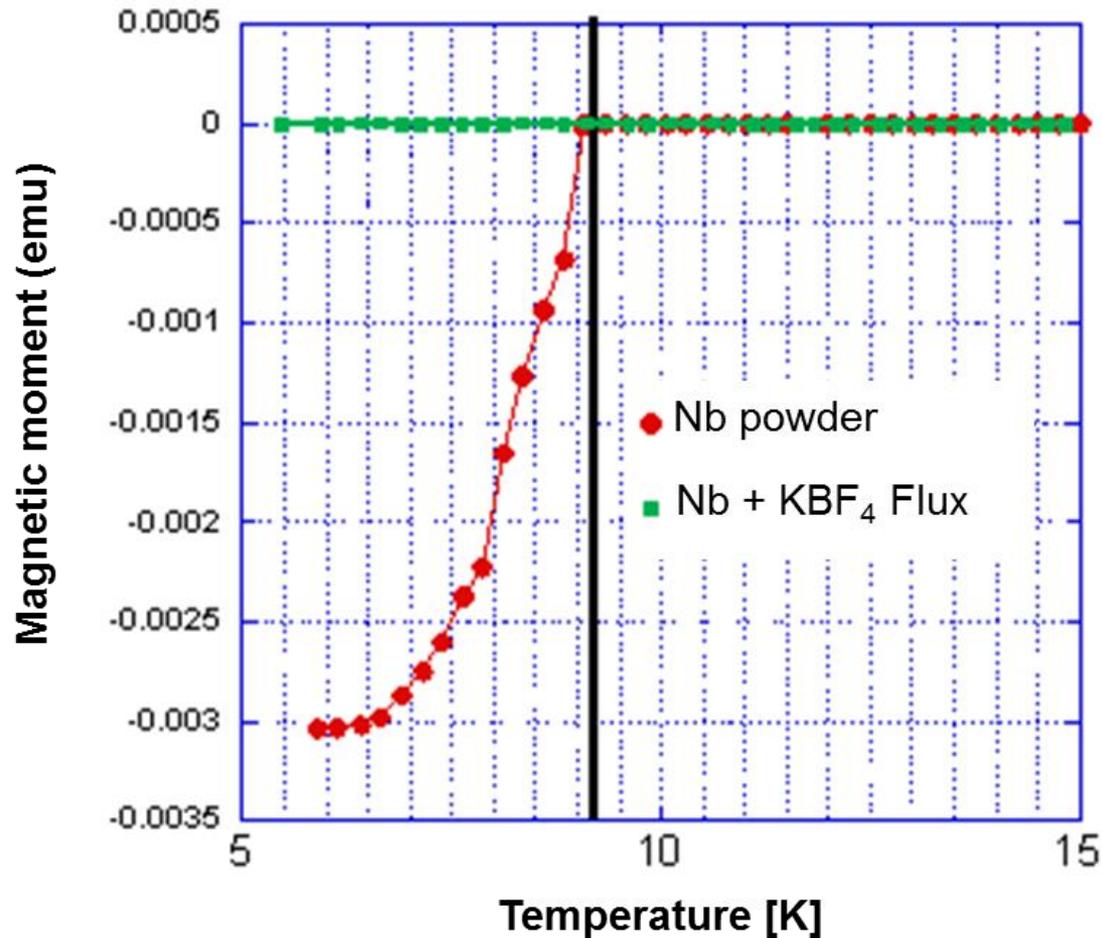


	T [°C]			
	700	750	800	850
Solubility, <i>K</i> [wt.%]	0.020	0.034	0.057	0.10
Dissolution rate constant, <i>K<sub>I</sub></i> [m/s]	4.60	5.10	6.20	6.80
Coefficient of diffusion, <i>D</i> ·10 <sup>9</sup> [m <sup>2</sup> /s]	1.61	1.86	2.49	2.89

$$C = C_s \left[ 1 - \exp\left(-\frac{k \cdot s \cdot t}{v}\right) \right]$$

Requires high temperature for larger Nb particles and high concentrations

# Addition of Nb fine metal powder to liquid Al



Nb- Superconductivity – 9K

To verify the Nb dissolution, magnetic moment vs temperature measured

<45  $\mu\text{m}$

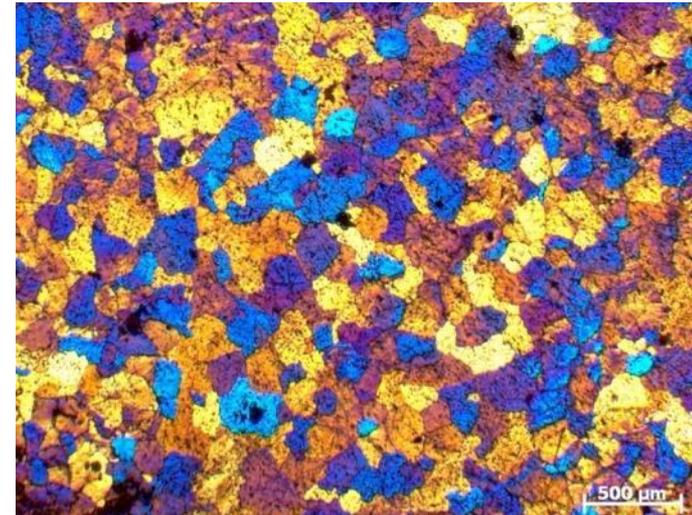
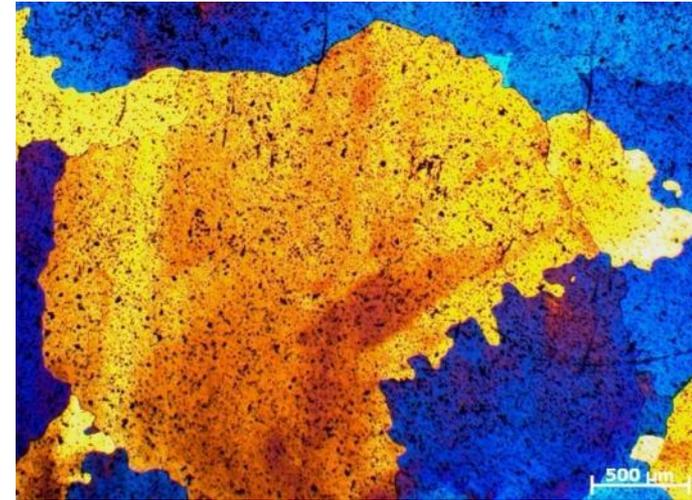
# EFFECT OF Nb on CP Al



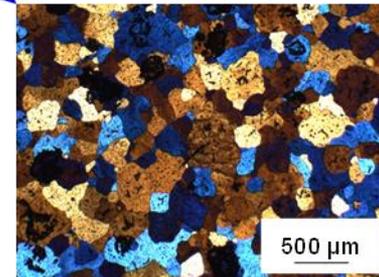
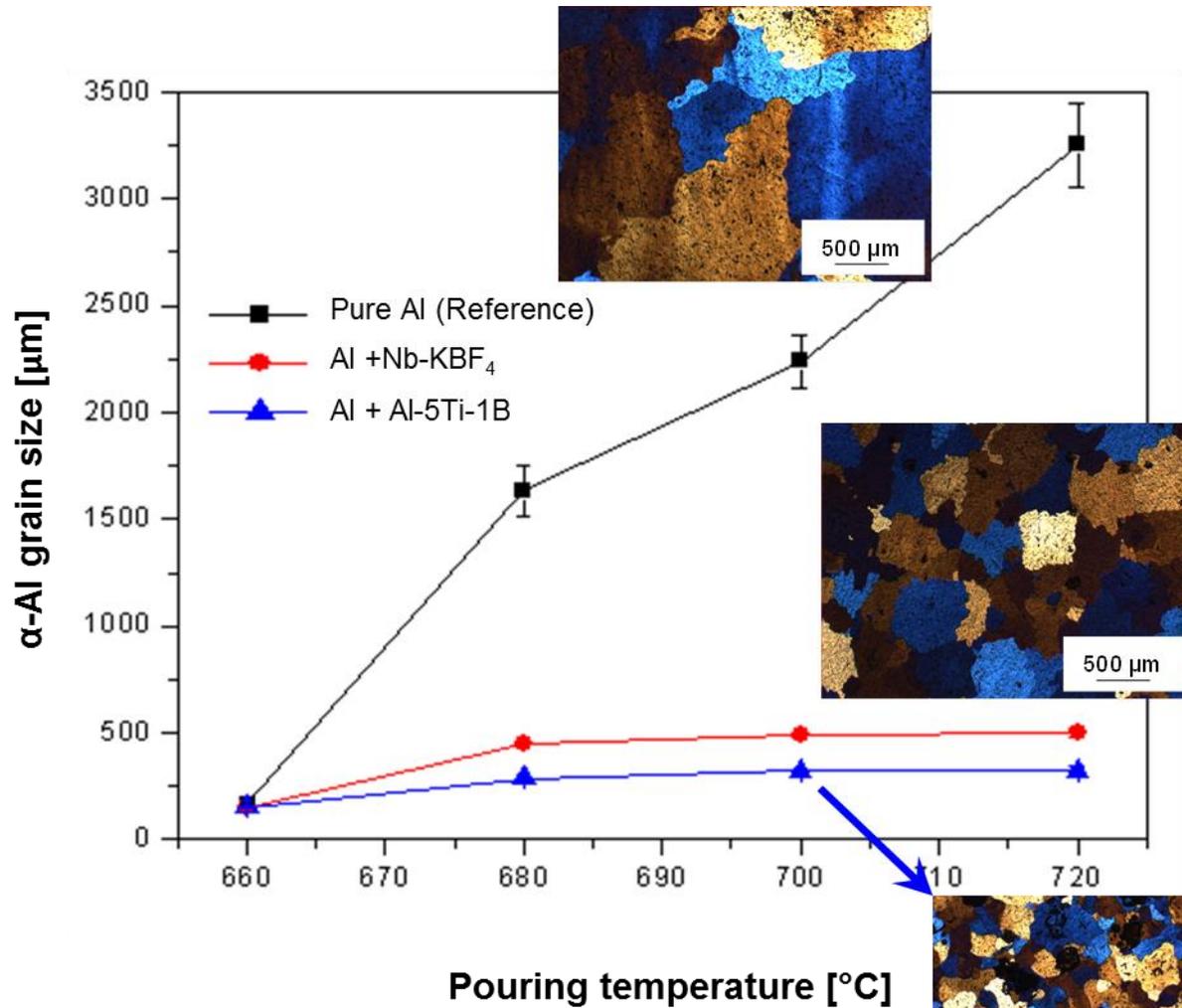
CP-Aluminium



Al with Nb-B

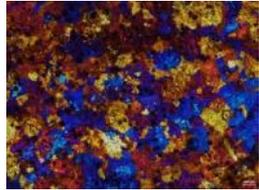


# COMPARISON OF Al-Ti-B AND Nb-B ON CP Al

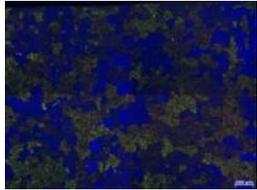


# COMPARISON OF Al-Ti-B AND Nb-B TO HYPOEUTECTIC BINARY Al-Si ALLOYS

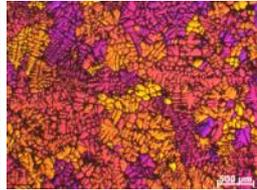
Al-1Si



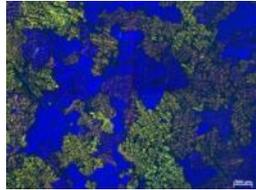
Al-2Si



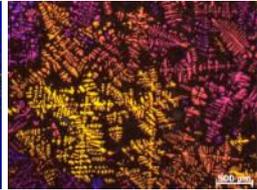
Al-4Si



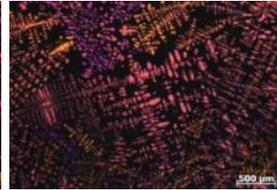
Al-5Si



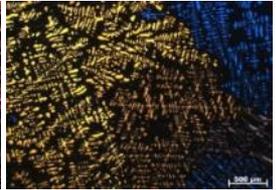
Al-6Si



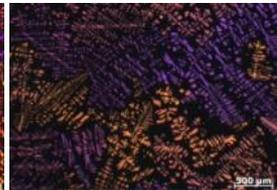
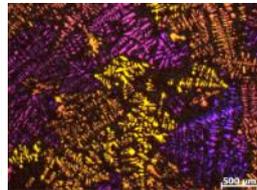
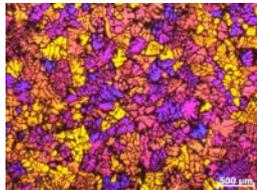
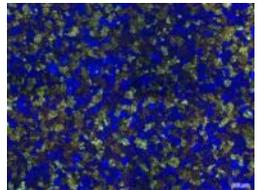
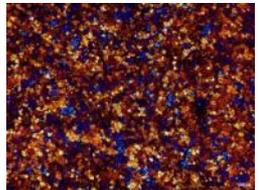
Al-8Si



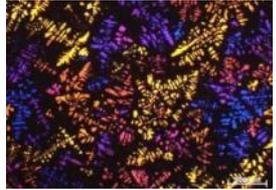
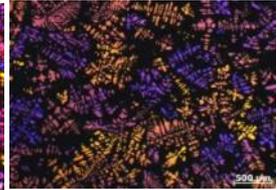
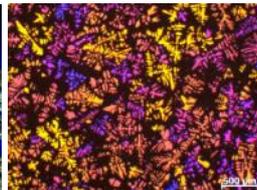
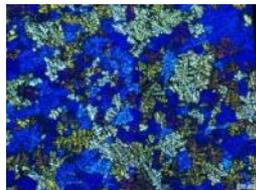
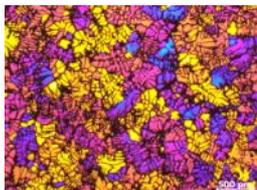
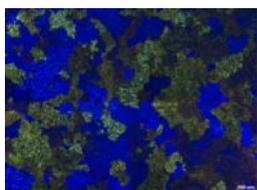
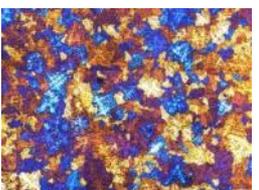
Al-10Si



Reference

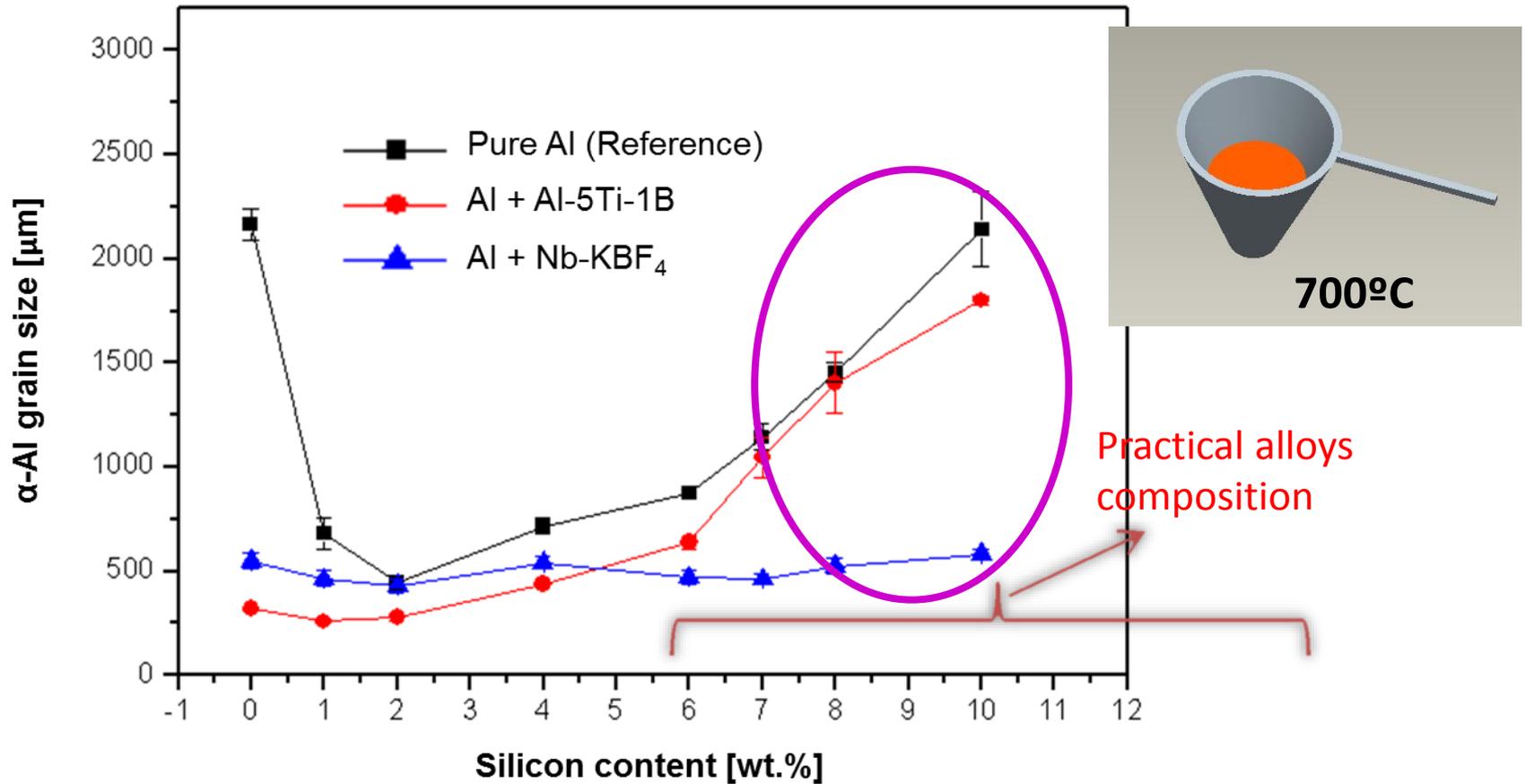


0.1wt.% Al-5Ti-1B



0.1wt.% Nb & B (powders)

# COMPARISON OF Al-Ti-B AND Nb-B TO HYPOEUTECTIC BINARY Al-Si ALLOYS



# Al-Si alloys for automotive applications

## Engine & transmission Components

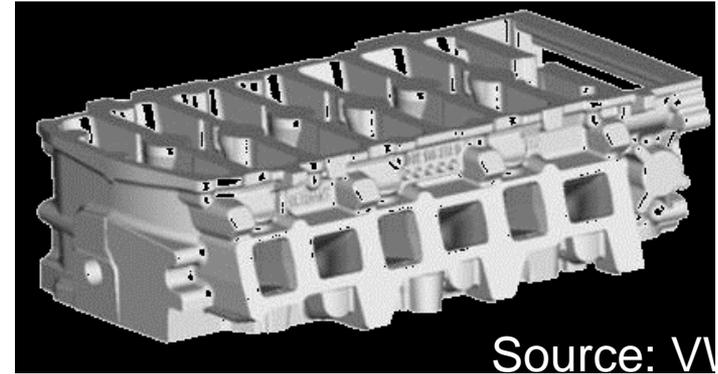
Crankcases

Cylinder heads

Intake manifolds

Housings manual/automatic transmissions

Housings power transfer units

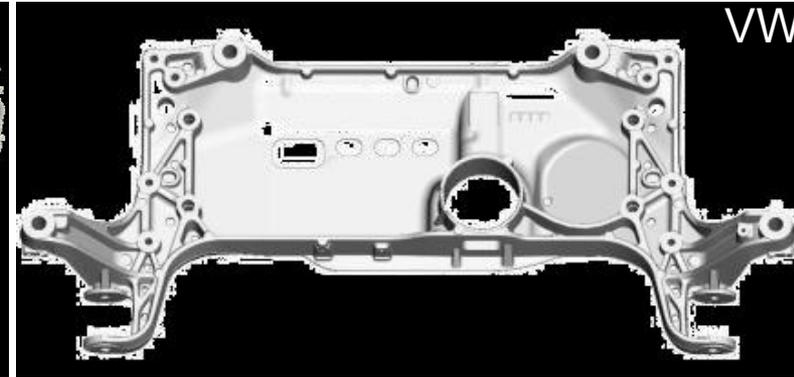


## Chassis Components

Subframes

Knuckles

Steering housings



## Structural Components

Body structures

Instrument panels

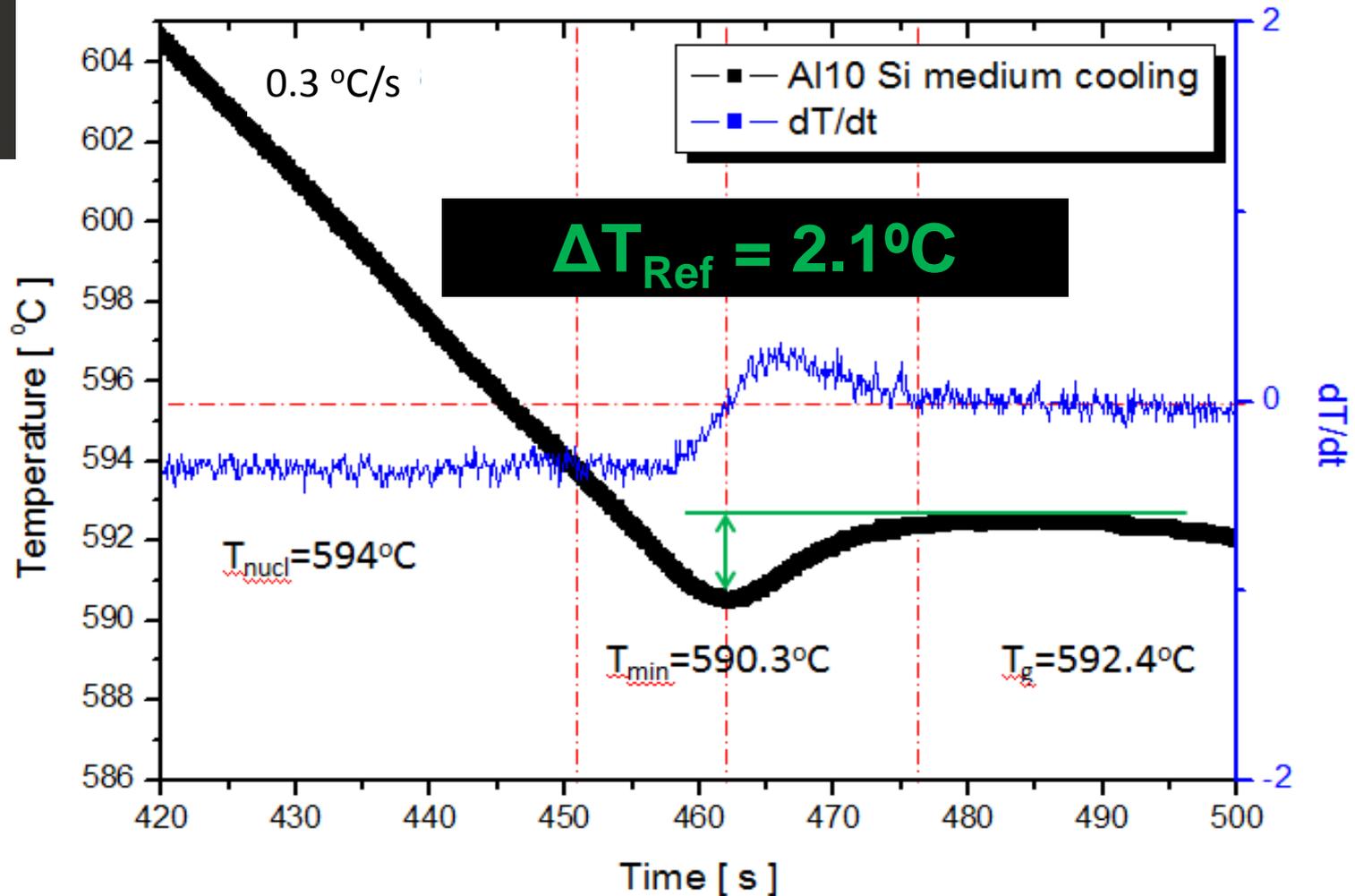
Door frames

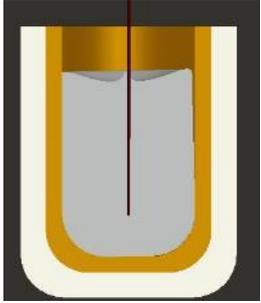
## Wheels



# Undercooling for Al-10Si alloy

## Al-10 Si alloy cooling curve





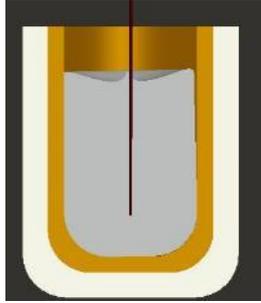
Reference

Grain size up to 1 cm

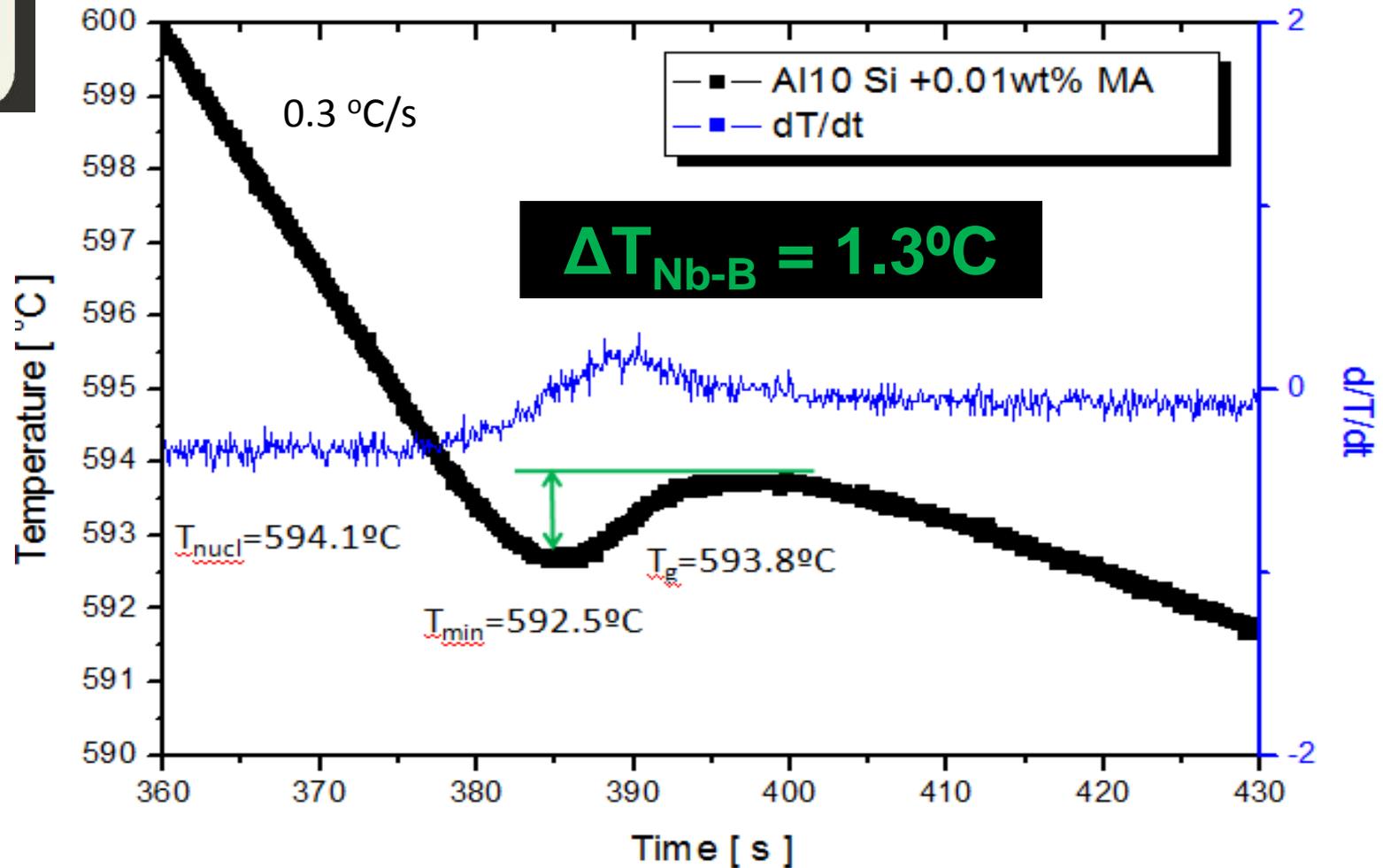


$$\Delta T_{\text{Ref}} = 2.1^{\circ}\text{C}$$

# Undercooling in the presence of $\text{NbB}_2/\text{Al}_3\text{Nb}$



Al-10 Si alloy with grain refiner



# GRAIN STRUCTURE

Reference Grain size up to 1 cm



$$\Delta T_{\text{Ref}} = 2.1^{\circ}\text{C}$$

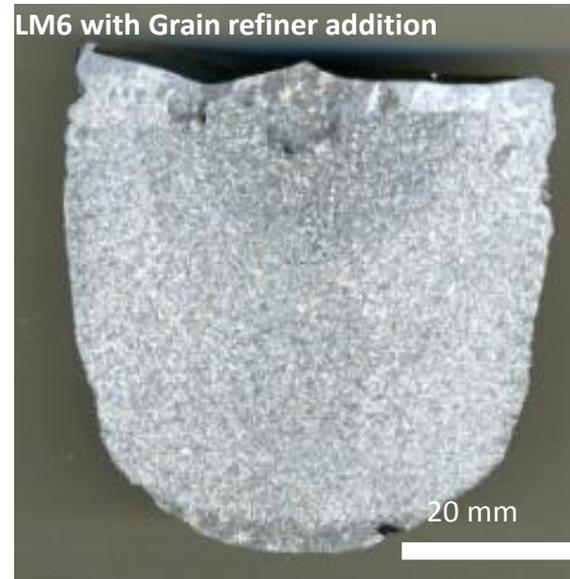
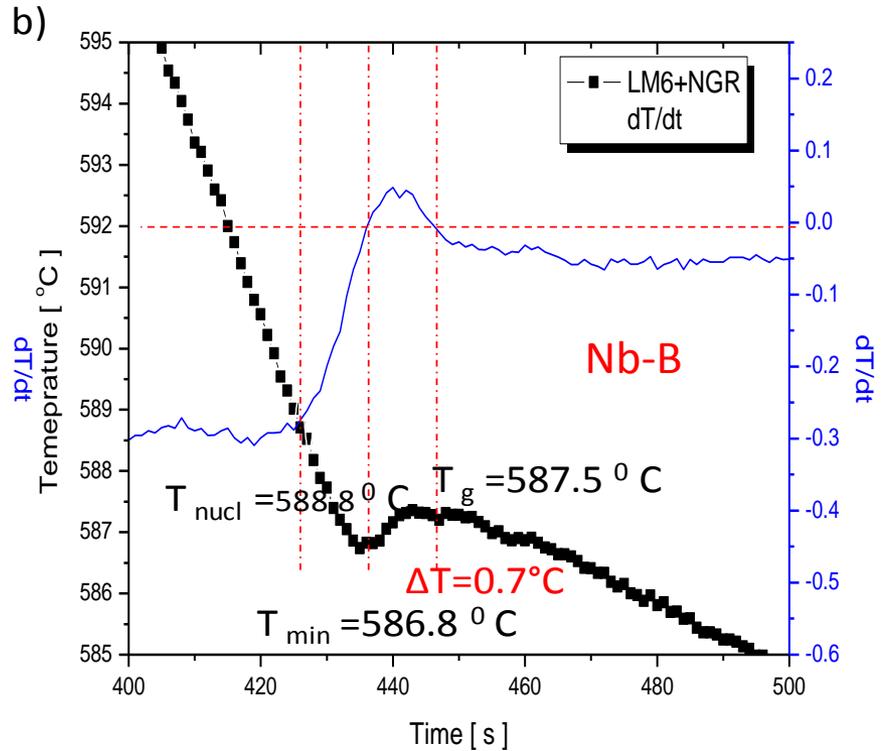
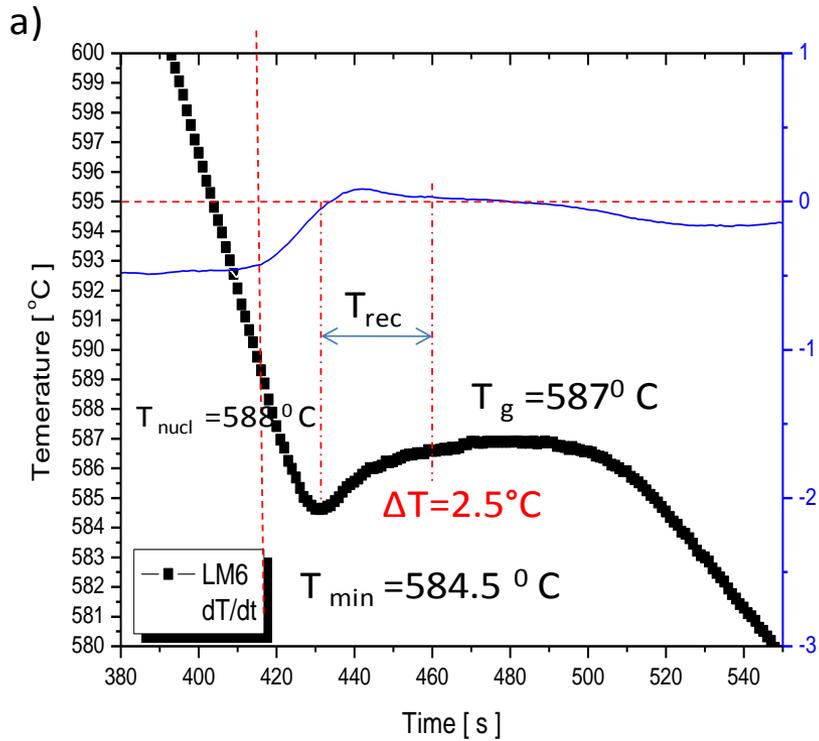
Nb-B

Grain size: 2-3 mm



$$\Delta T_{\text{Nb-B}} = 1.3^{\circ}\text{C}$$

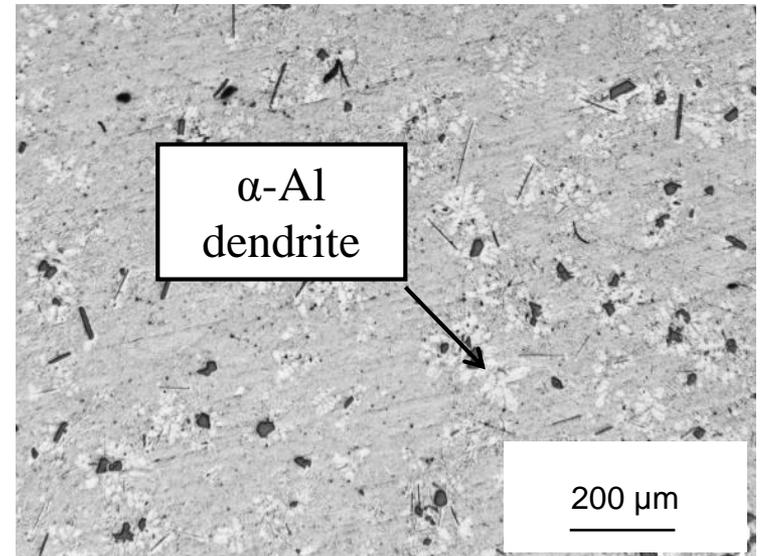
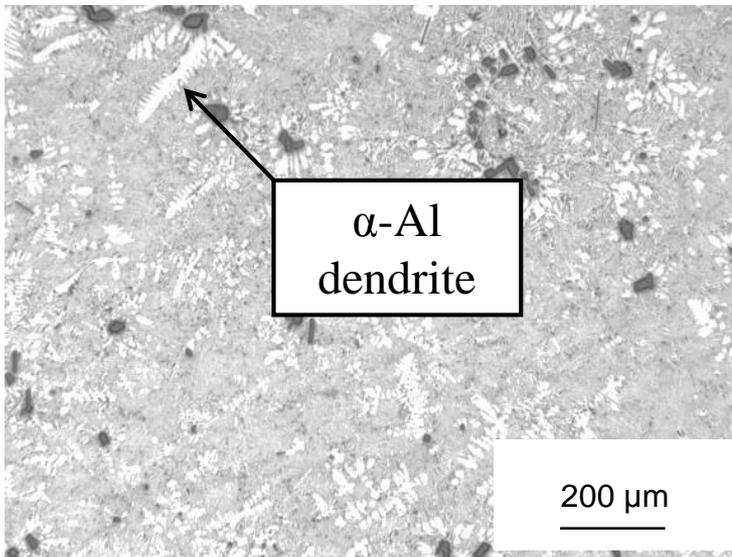
# Cooling curves for Al-11Si (LM6) alloy



# HYPEREUTECTIC BINARY Al-Si ALLOYS

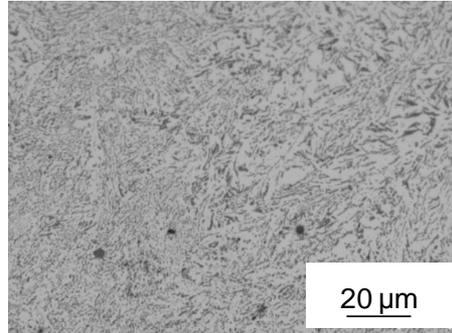
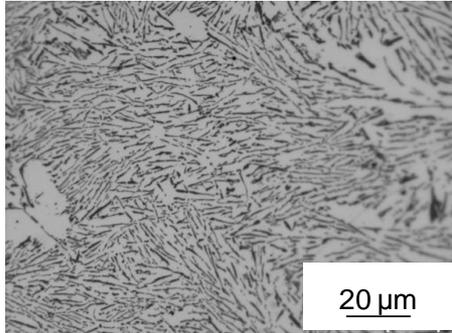


Al-14Si

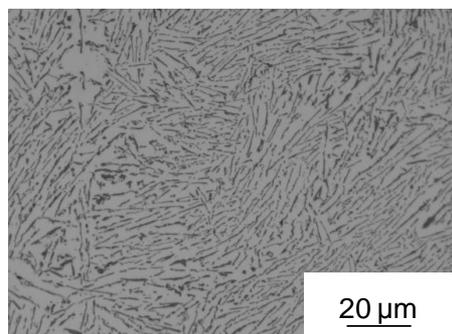
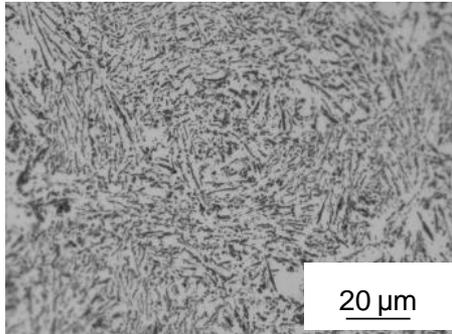


# HYPEREUTECTIC BINARY Al-Si ALLOYS - EUTECTIC

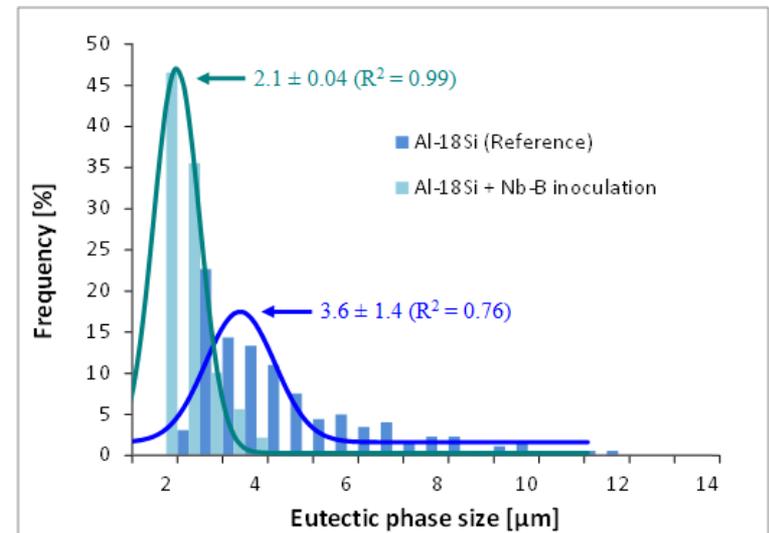
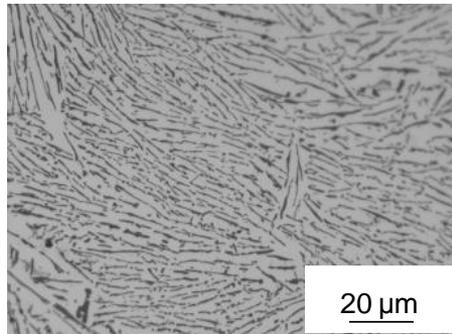
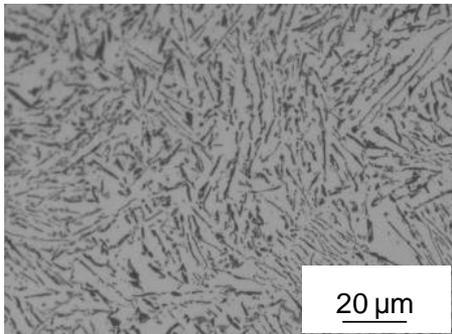
Al-16Si



Al-18Si

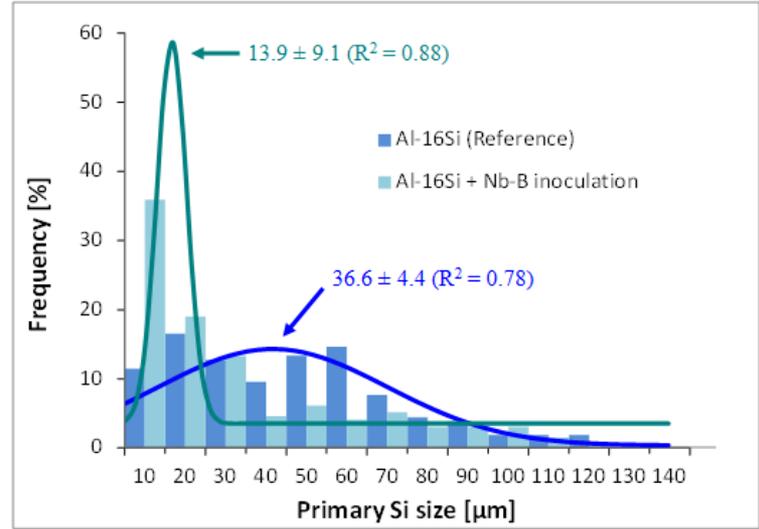
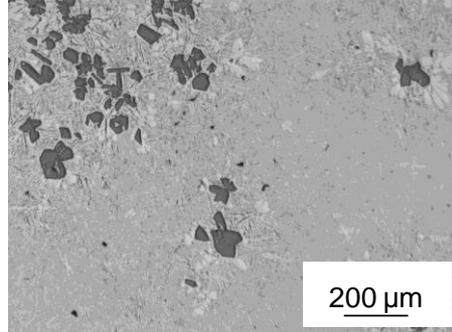
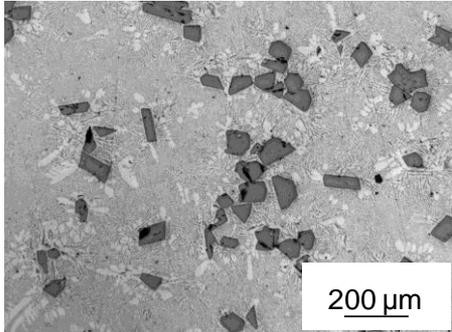


Al-27Si

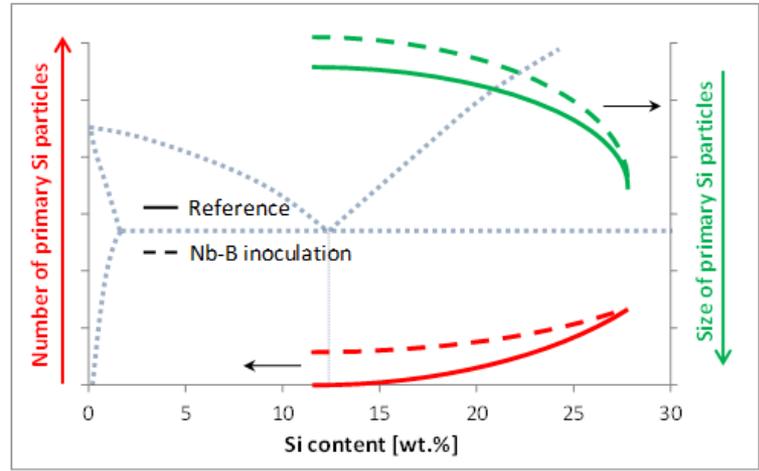
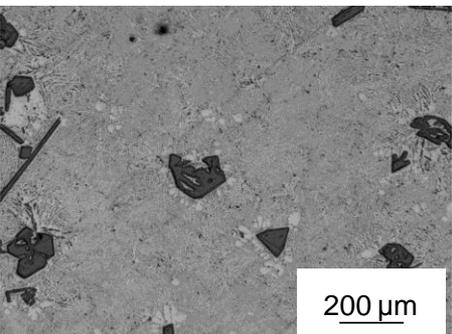
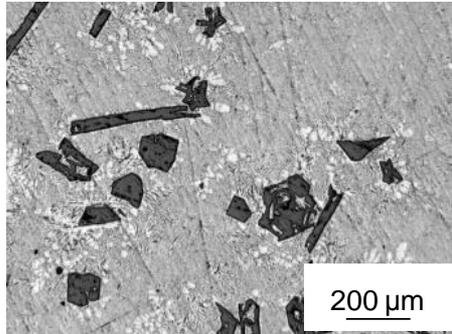


# HYPEREUTECTIC BINARY Al-Si ALLOYS – PRIMARY SI

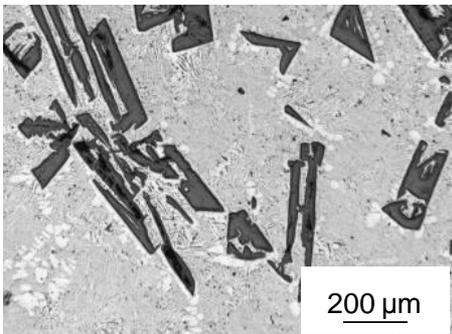
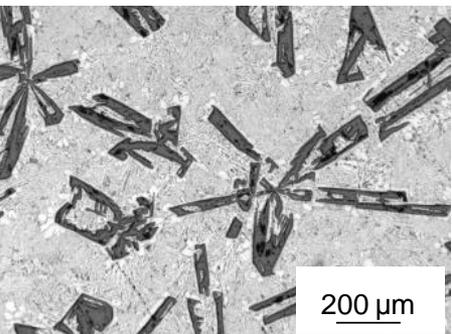
Al-16Si



Al-18Si



Al-27Si



# Application of Nb-B grain refiner to Al-Si commercial alloys

# Commercial alloys tested with Nb-B

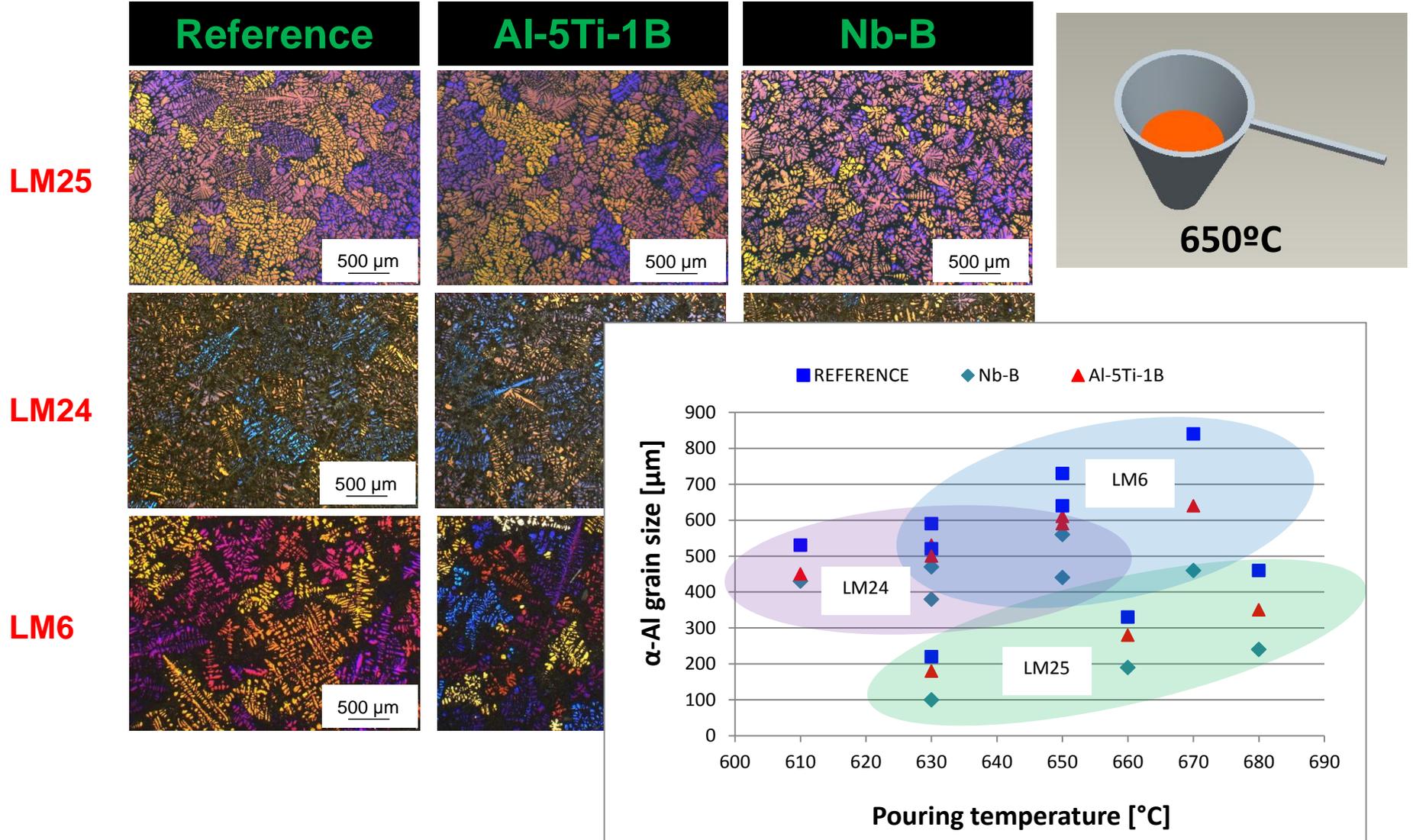
GB	USA	Si	Mg	Mn	Cu	Ni	Zn	Fe
LM6	A413	10.0-13.0	0.1max	0.5max	0.1max	0.1max	0.1max	0.6max
LM13	336	10.0-13.0	0.2-0.4	0.5max	0.7-1.5	1.5max	0.1max	1max
LM24	A380	7.5-9.5	3 max	0.5max	3.0-4.0	0.5	3	1.3max
LM25	A356	6.5-7.5	0.2-0.6	0.3	0.2	0.1	0.1	0.5
		9.99	0.005	0.005	0.0017	0.0044	0.005	0.09
		10.98	0.268	0.21	2.134	0.068	0.778	0.83
		6.06	0.275	0.265	2.725	0.0257	0.305	0.356
		11.9	0.8	0.005	3.7	2	0.003	0.12

# Nb-B Grain Refiner for Al-Si cast alloys

## Highly effective for Al-Si alloys & Mg alloys

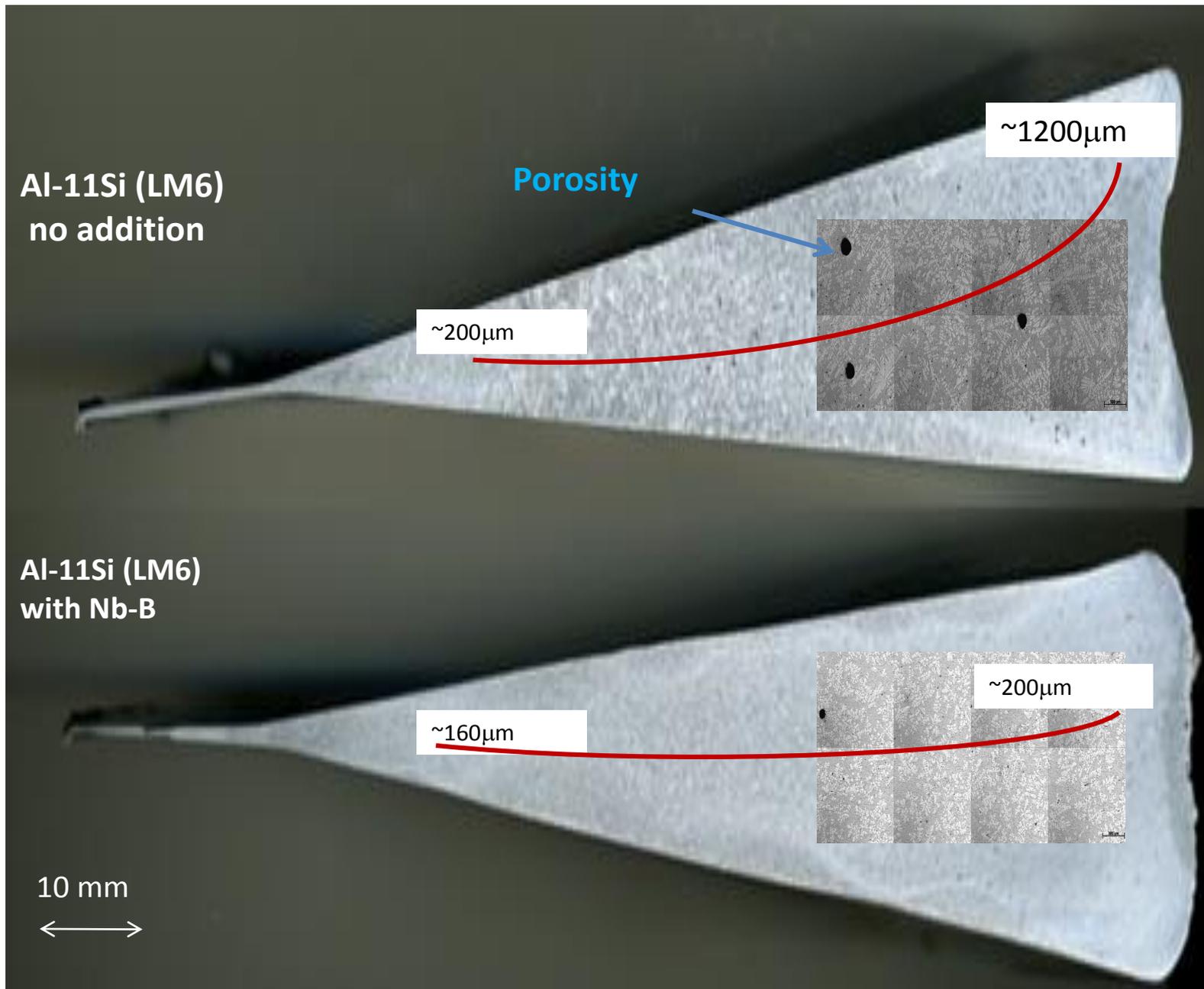
- Fine & uniform grain structure
- Grain size is less sensitive to cooling rate
- Highly effective in sand casting cooling conditions
- Reduced porosity & macro defects
- Fine eutectic structure & intermetallics
- Improved ductility & strength
- Tolerant to Fe contamination
- Recycling of Al-Si scrap

# COMMERCIAL HYPOEUTECTIC Al-Si ALLOYS



# Nb-B Grain Refiner for Al-Si cast alloys

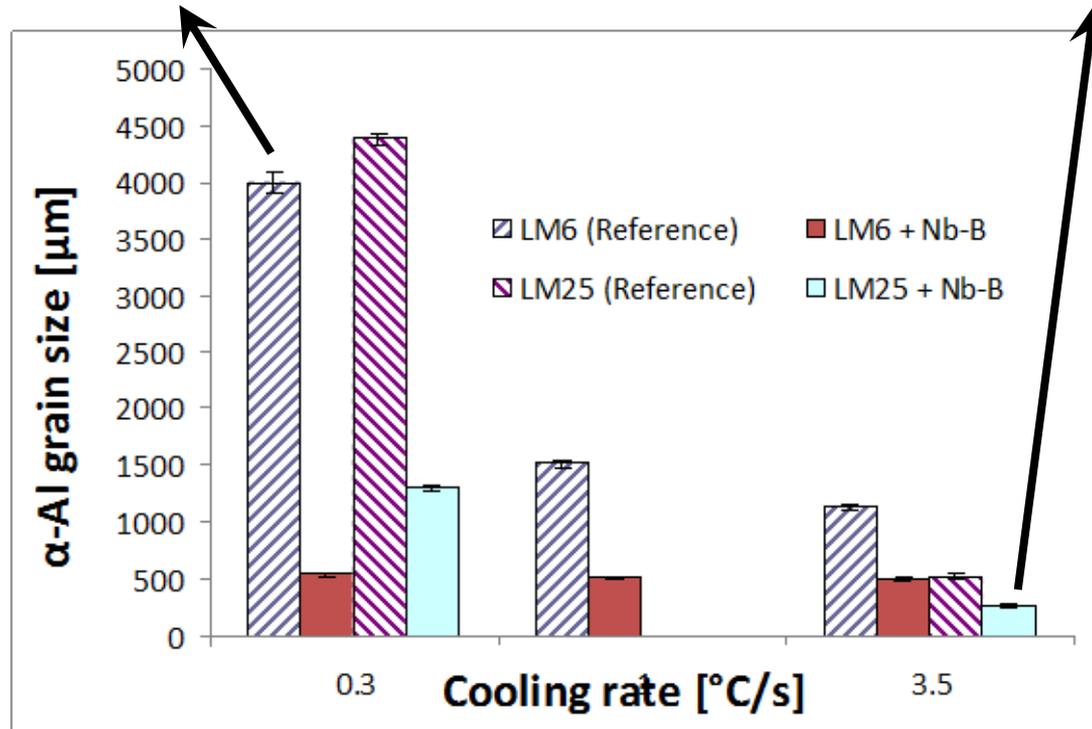
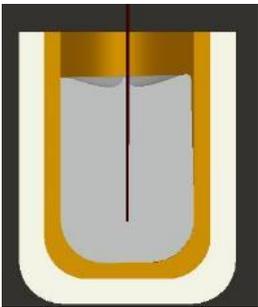
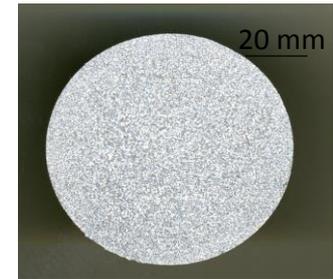
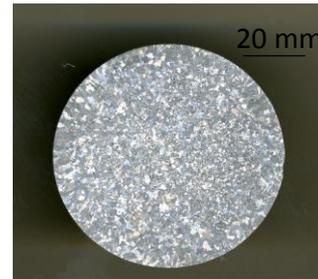
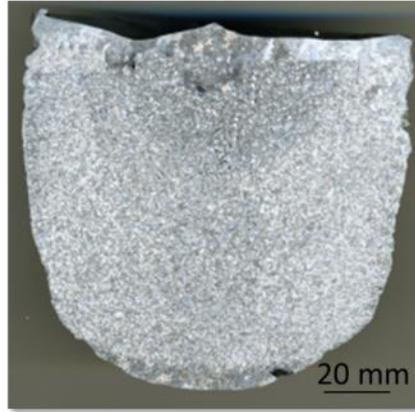
- ❑ Highly effective for Al-Si alloys & Mg alloys
- ❑ **Fine & uniform grain structure**
- ❑ Grain size is less sensitive to cooling rate
- ❑ Highly effective in sand casting cooling conditions
- ❑ Reduced porosity & macro defects
- ❑ Fine eutectic structure & intermetallics
- ❑ Improved ductility & strength
- ❑ Tolerant to Fe contamination
- ❑ Recycling of Al-Si scrap



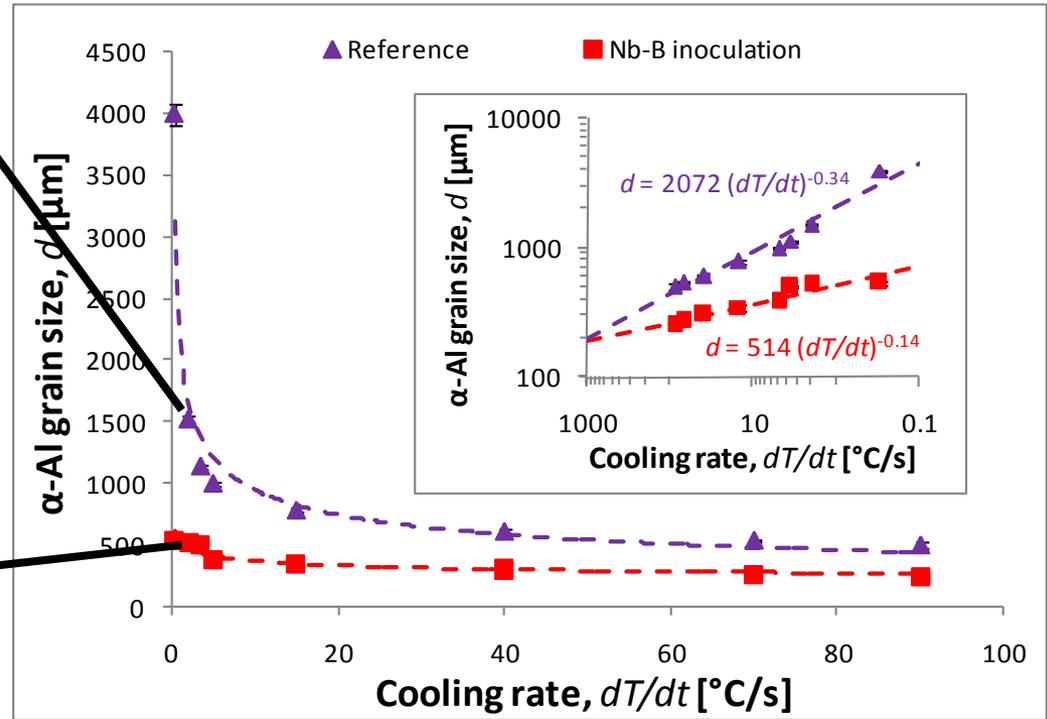
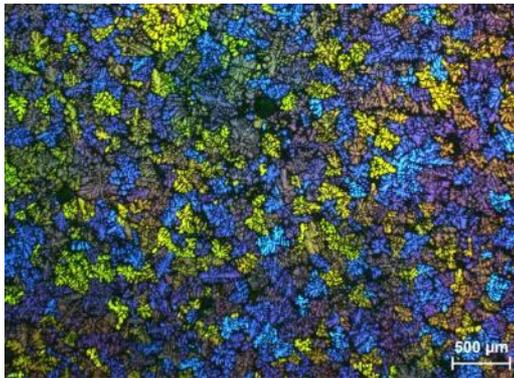
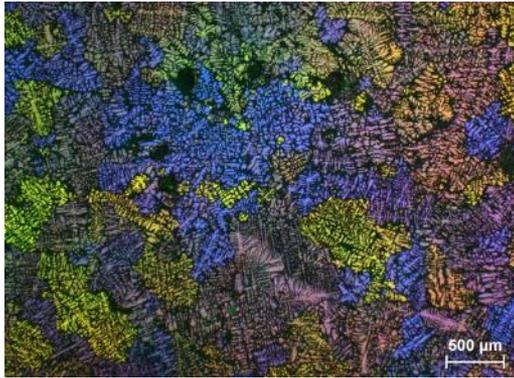
# Al-Nb-B Grain Refiner for Al-Si cast alloys

- Highly effective for Al-Si alloys & Mg alloys
- Fine & uniform grain structure
- Grain size is less sensitive to cooling rate**
- Highly effective in sand casting cooling conditions
- Reduced porosity & macro defects
- Fine eutectic structure & intermetallics
- Improved ductility & strength
- Tolerant to Fe contamination
- Recycling of Al-Si scrap

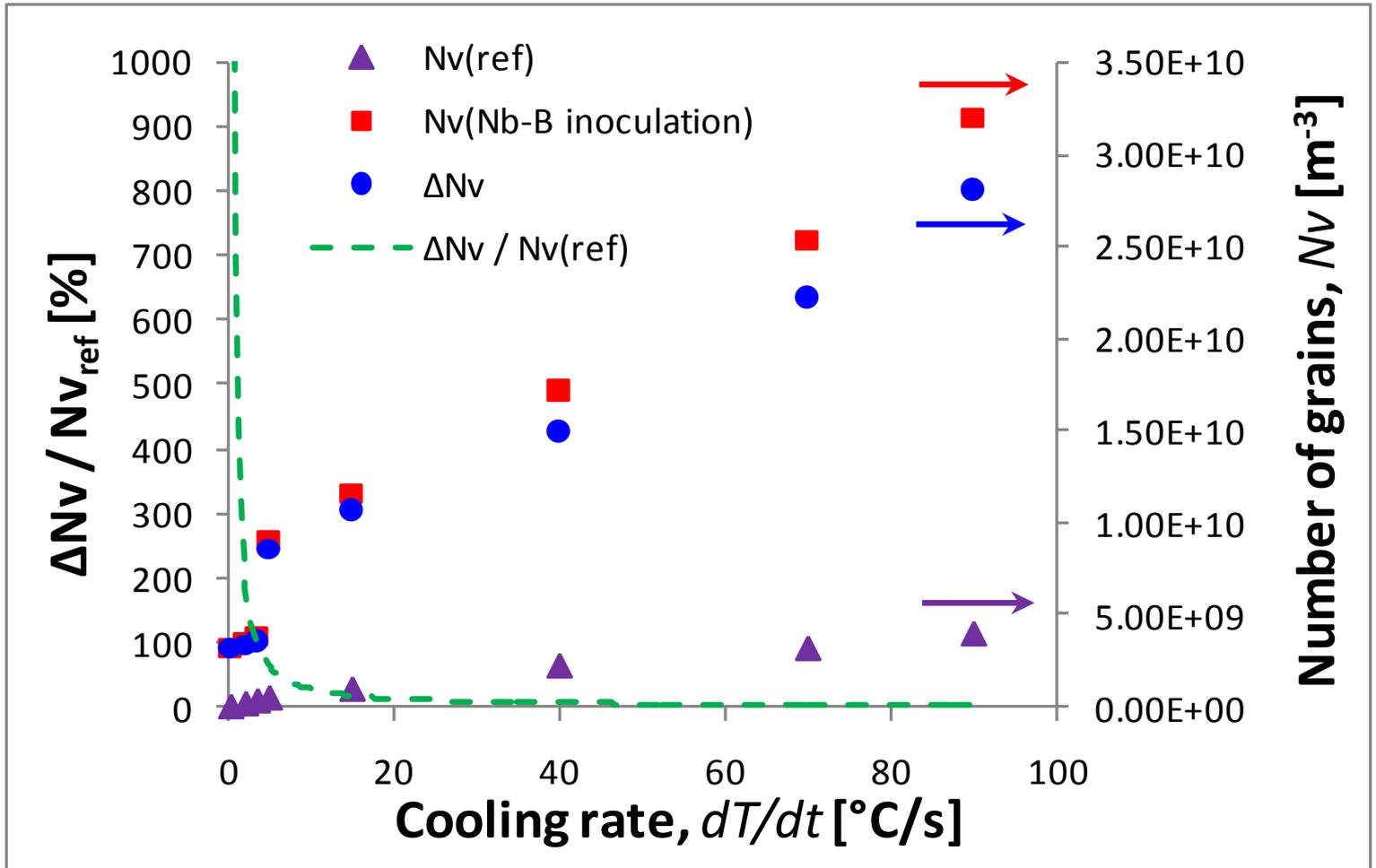
# EFFECT OF COOLING RATE



# EFFECT OF COOLING RATE



# EFFECT OF COOLING RATE



# Nb-B Grain Refiner for Al-Si cast alloys

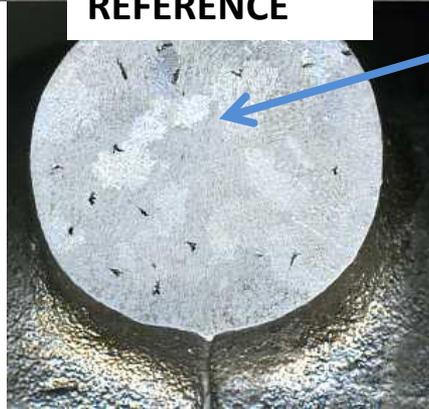
- ❑ Highly effective for Al-Si alloys & Mg alloys
- ❑ Fine & uniform grain structure
- ❑ Grain size is less sensitive to cooling rate

## ❑ Highly effective in sand casting cooling conditions

- ❑ Reduced porosity & macro defects
- ❑ Fine eutectic structure & intermetallics
- ❑ Improved ductility & strength
- ❑ Tolerant to Fe contamination
- ❑ Recycling of Al-Si scrap



**REFERENCE**

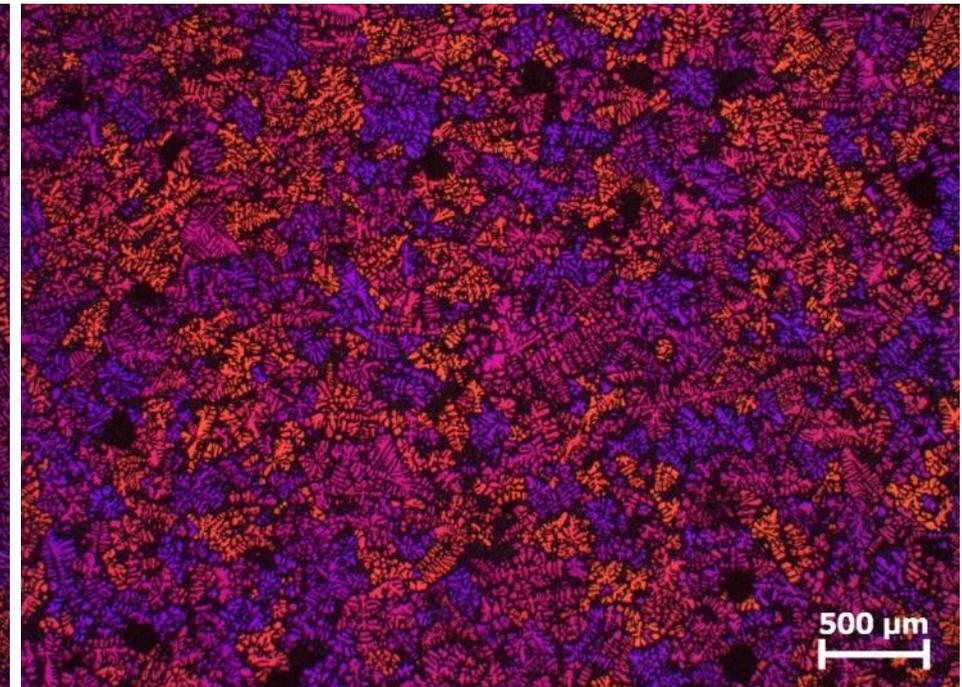
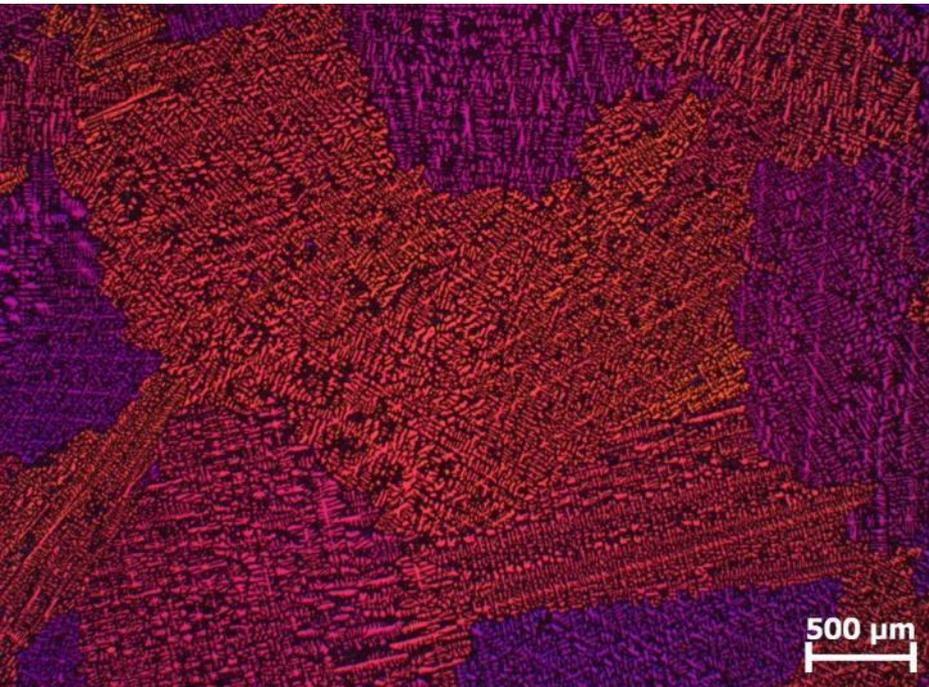


**Al-Nb-B  
ADDITION**



Al-9Si-2Cu-0.7Mg-0.15Fe

# A354

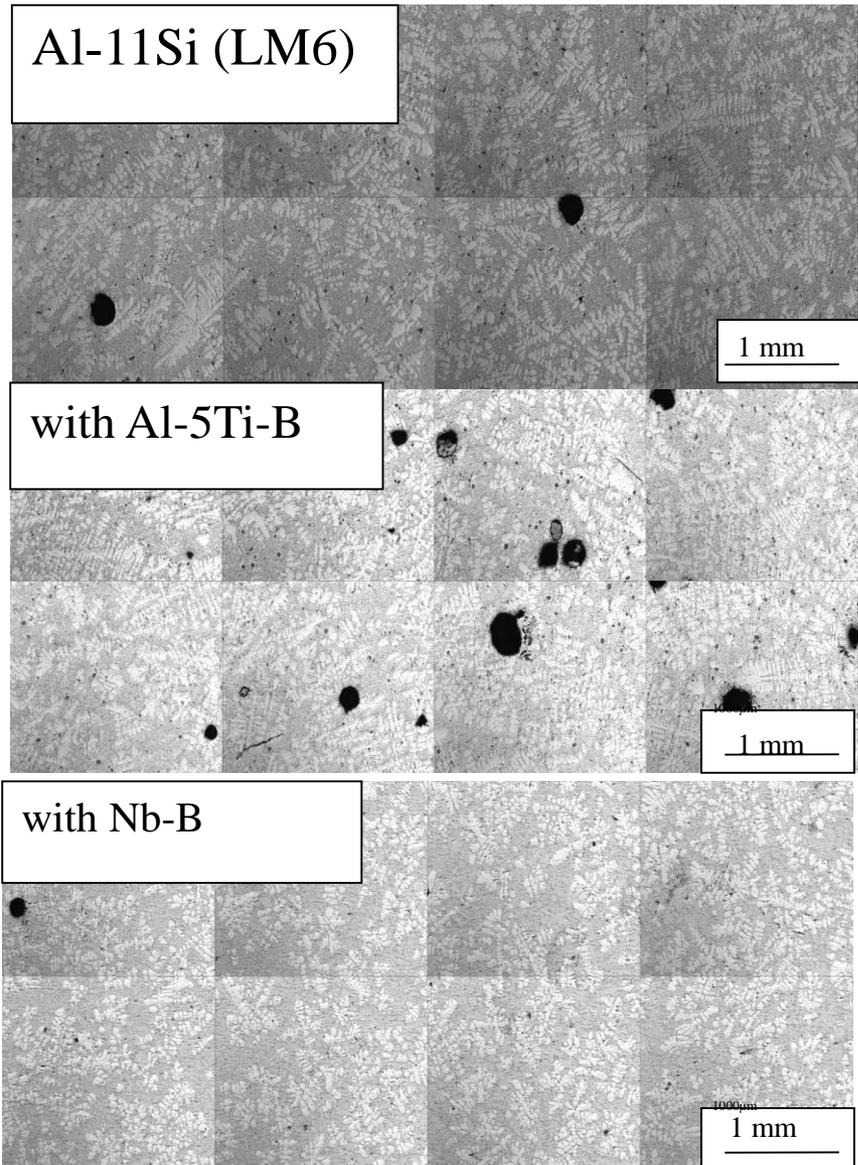


Alloy	Condn.	%Cu	%Mg	%Si	%Fe	%Mn	%Ni	%Zn	%Pb	%Sn	%Ti	%Sr
A354	CAST	1.60-2.0	0.50-0.60	8.6-9.44	0.154	0.05-0.10	0.054	0.10	0.014	0.054	0.10-0.154	0.02-0.030

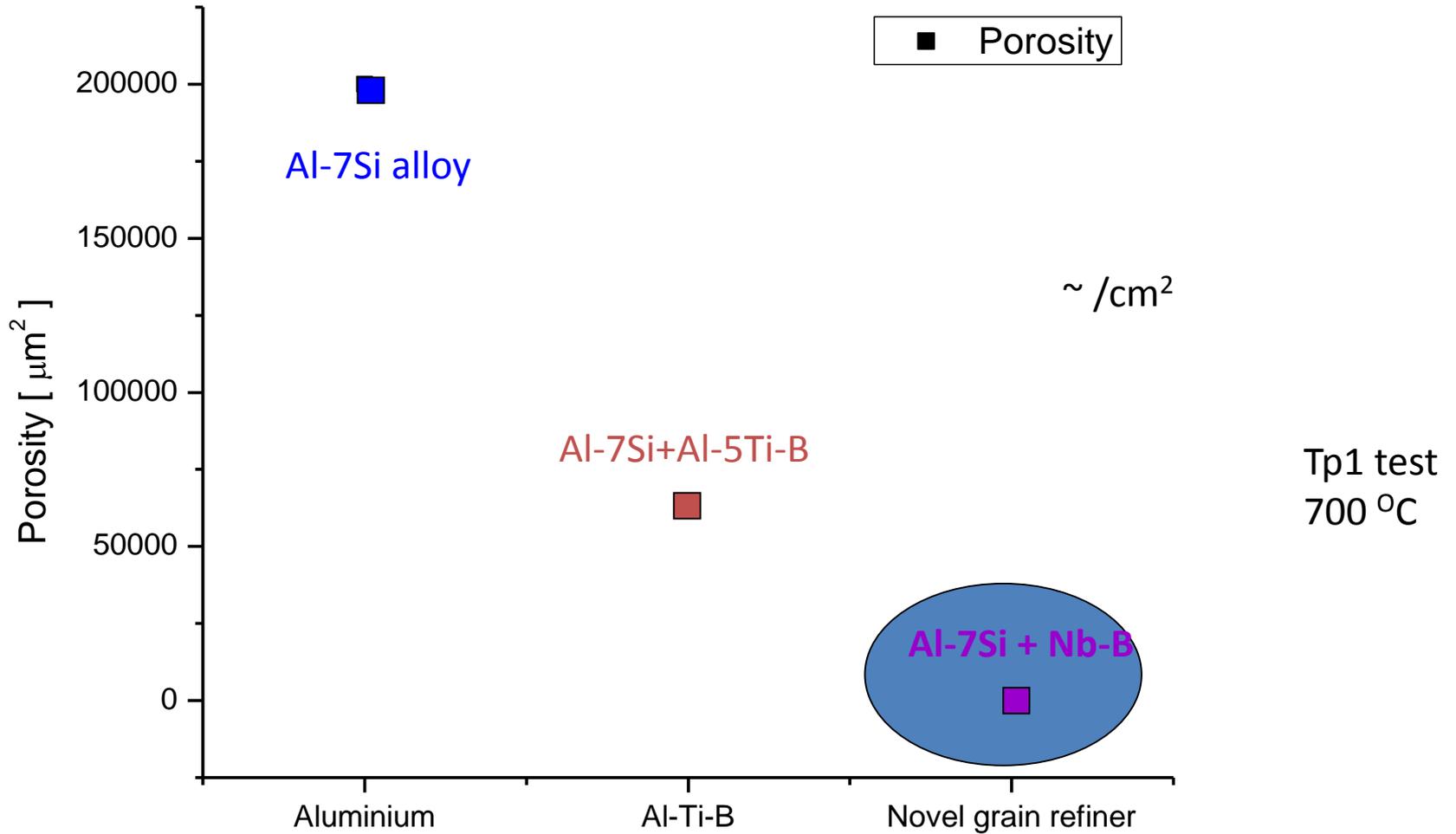
# Al-Nb-B Grain Refiner for Al-Si cast alloys

- Highly effective for Al-Si alloys & Mg alloys
- Fine & uniform grain structure
- Grain size is less sensitive to cooling rate
- Highly effective in sand casting cooling conditions
- Reduced porosity & macro defects**
- Fine eutectic structure & intermetallics
- Improved ductility & strength
- Tolerant to Fe contamination
- Recycling of Al-Si scrap

# Reduced Macro-porosity with Nb-B



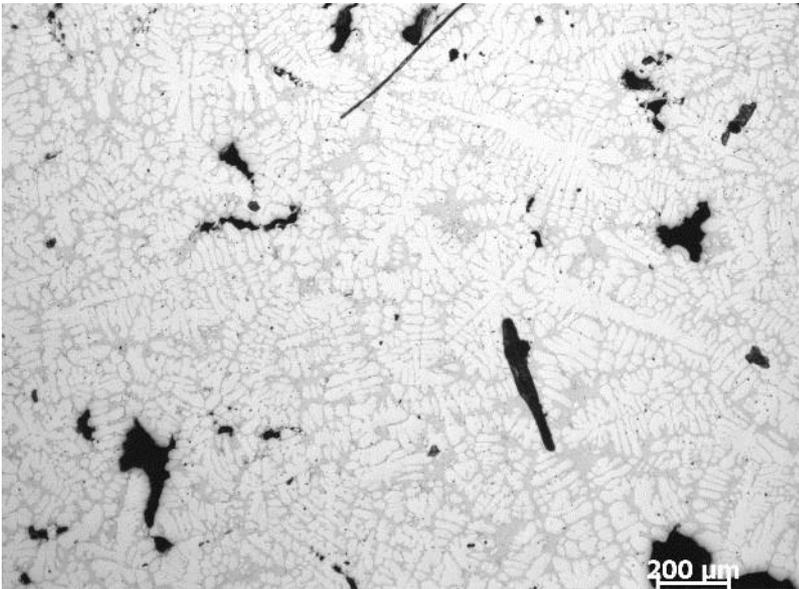
# Porosity



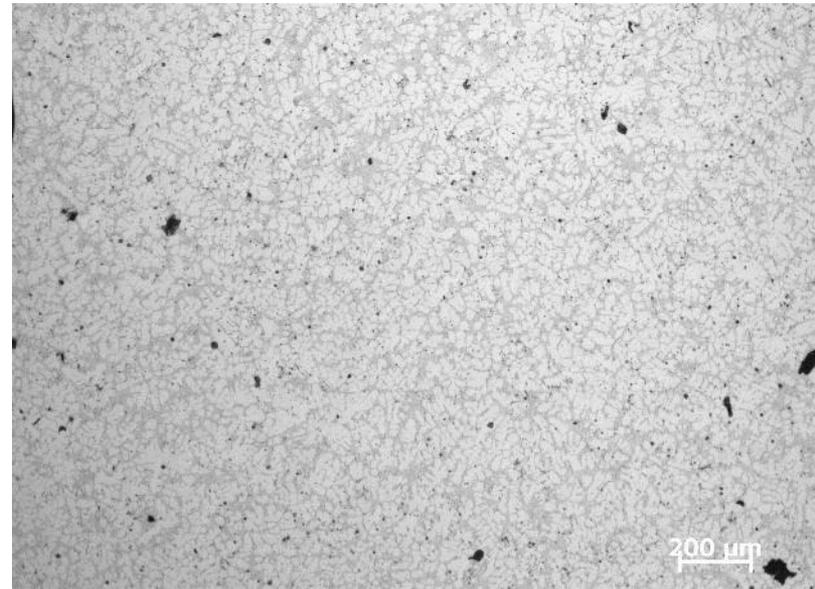
Reduced porosity in Nb-B grain refiner added castings

# Al-7Si alloy

Without



With Nb-B  
addition

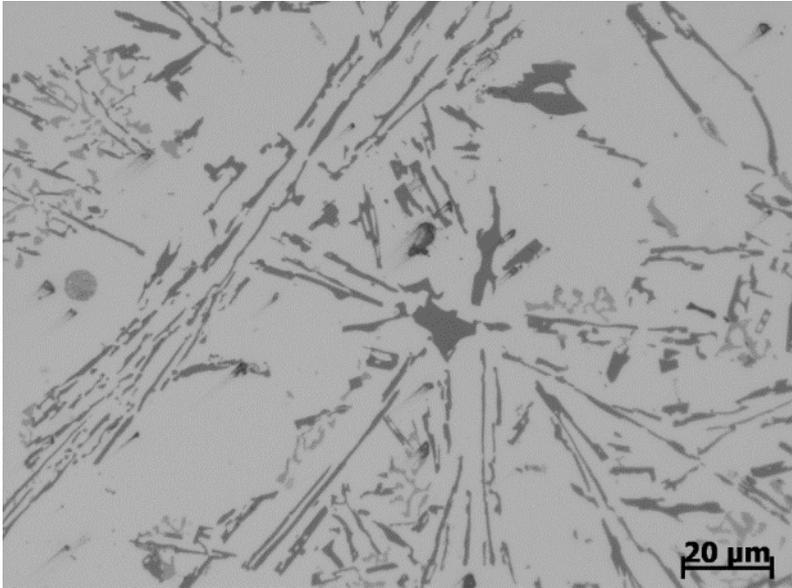


Fine grain structure  
Reduced porosity

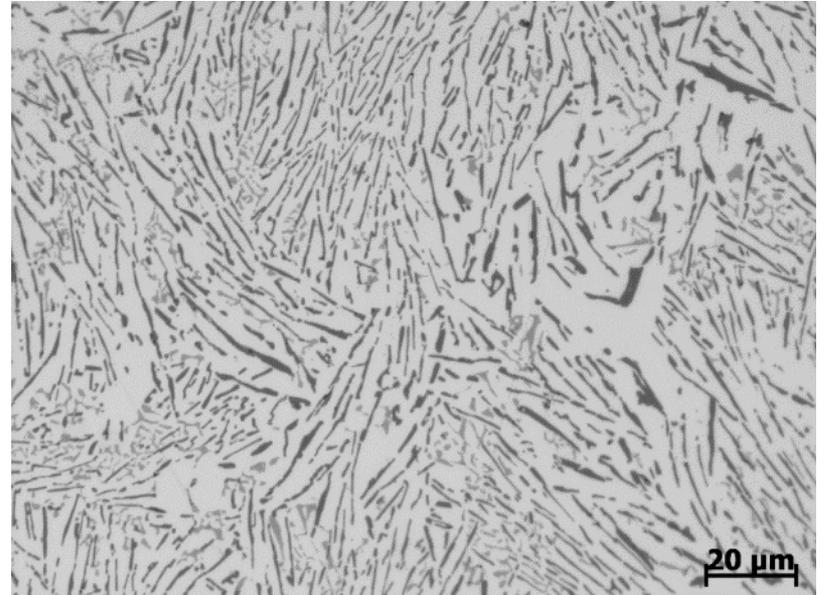
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- ❑ Improved ductility & strength
- ❑ Tolerant to Fe contamination
- ❑ Recycling of Al-Si scrap

# Fine eutectic structure

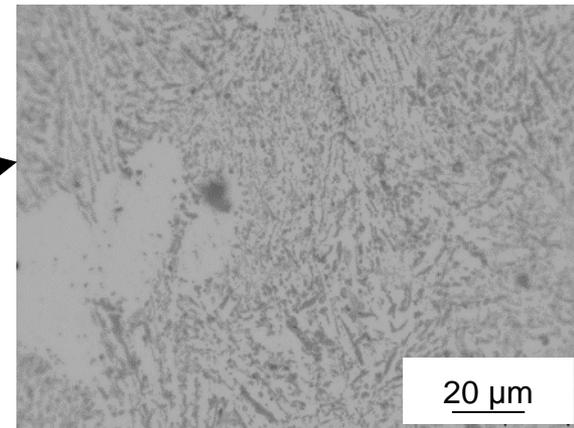
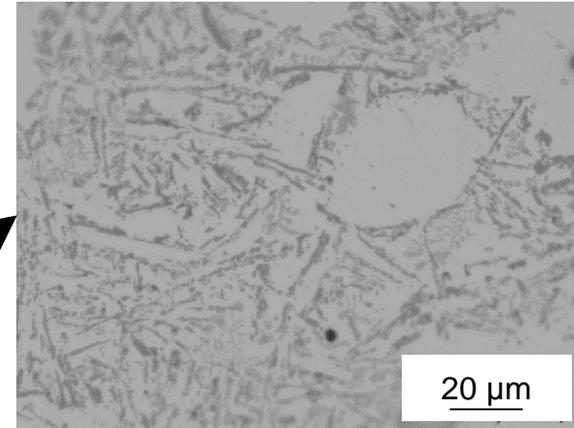
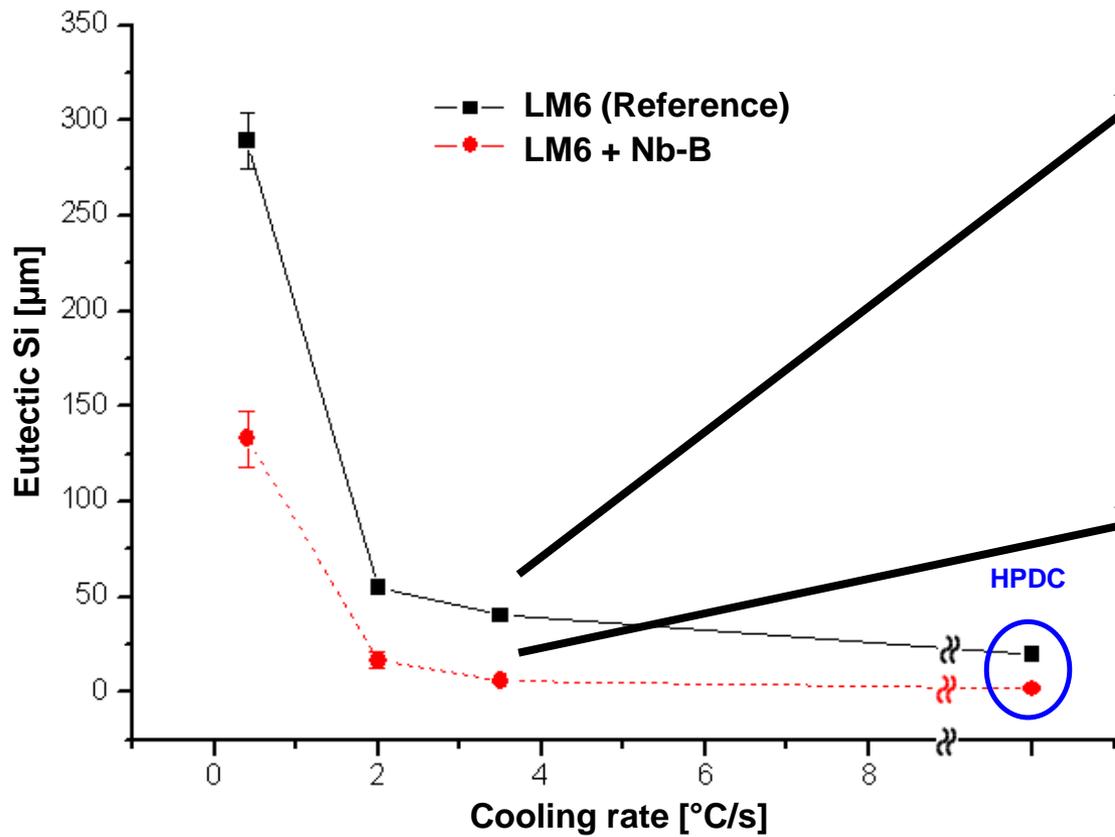


Al-11Si (LM6)



with Nb-B

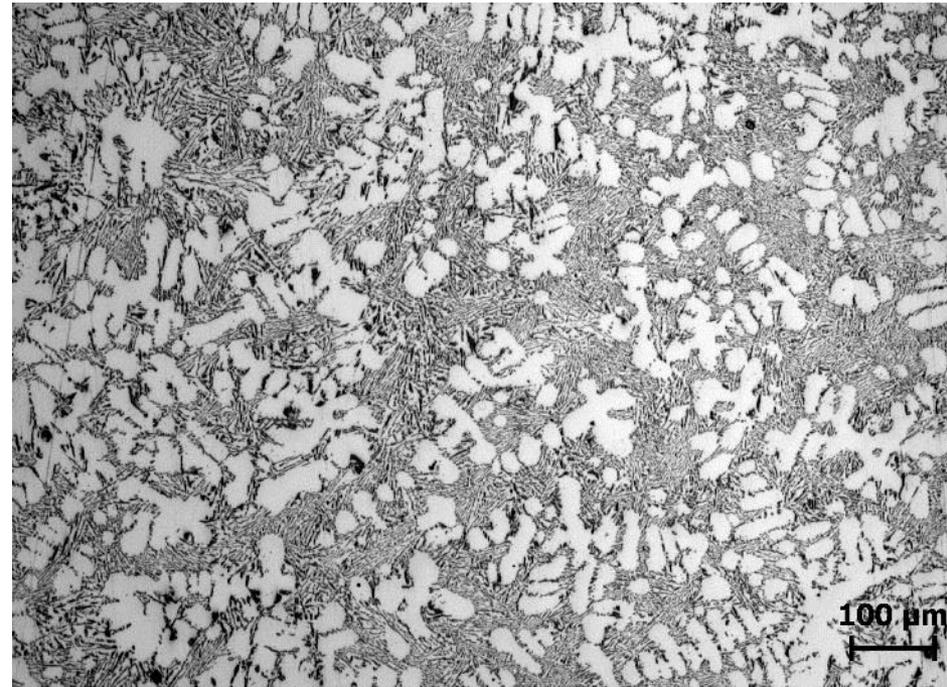
# Finer Eutectic Si - wider range of cooling rates



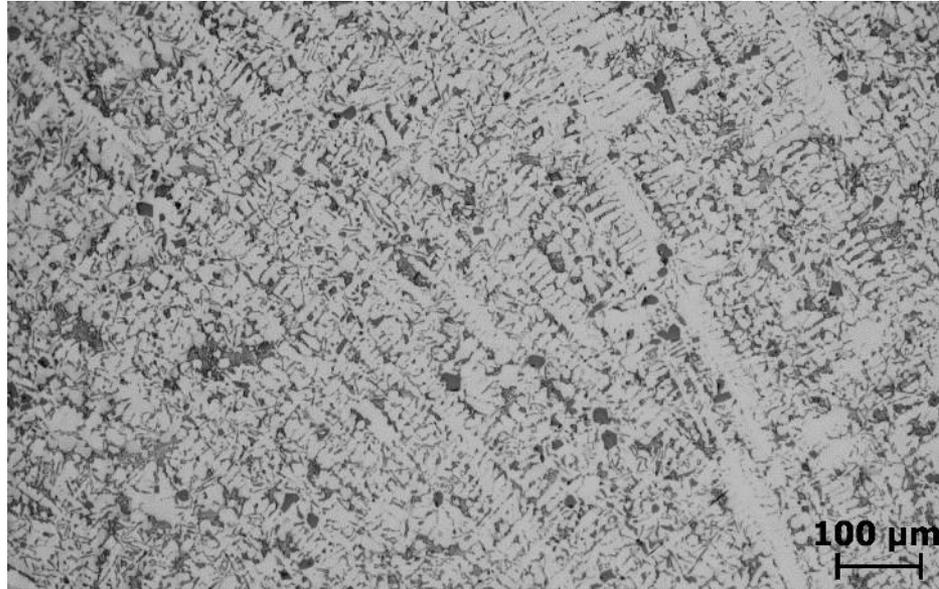
Al-10Si



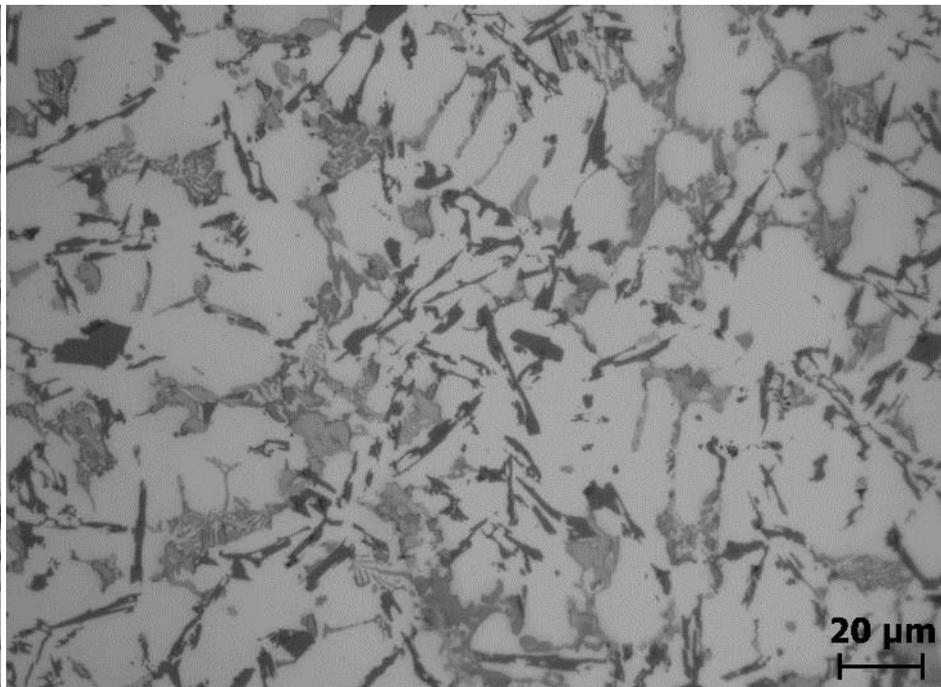
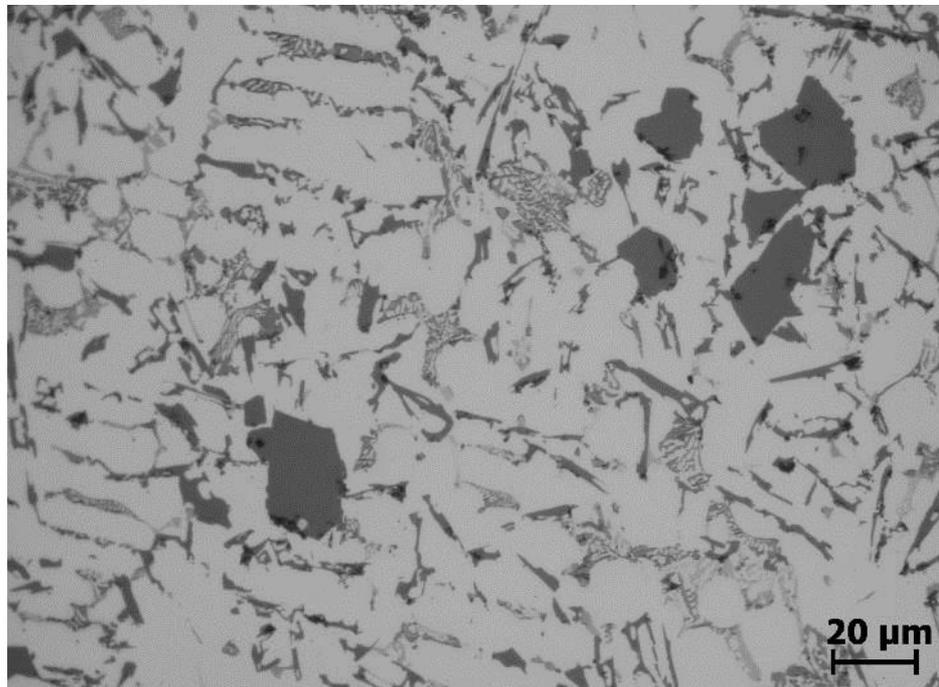
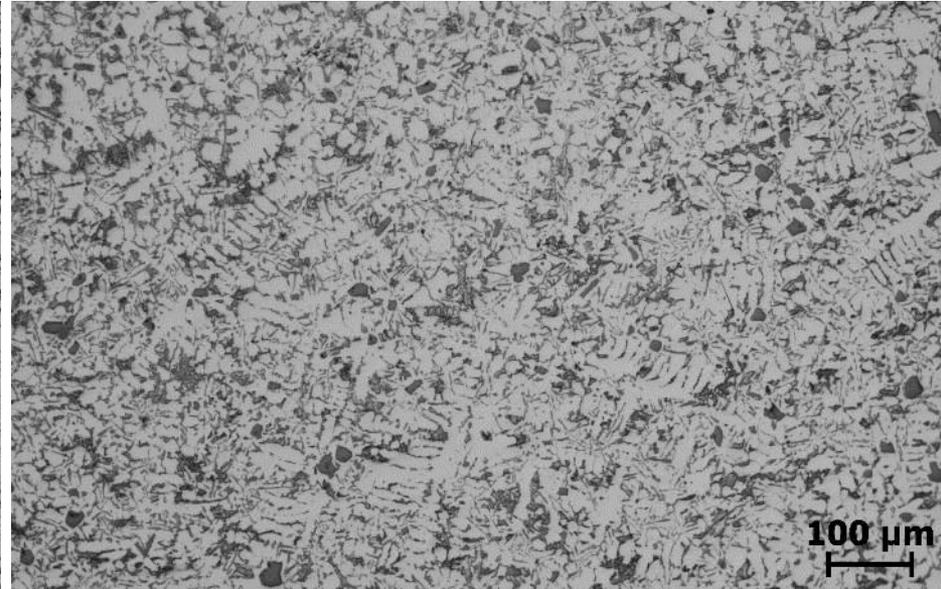
With Nb-B



Reference (Al-13Si)



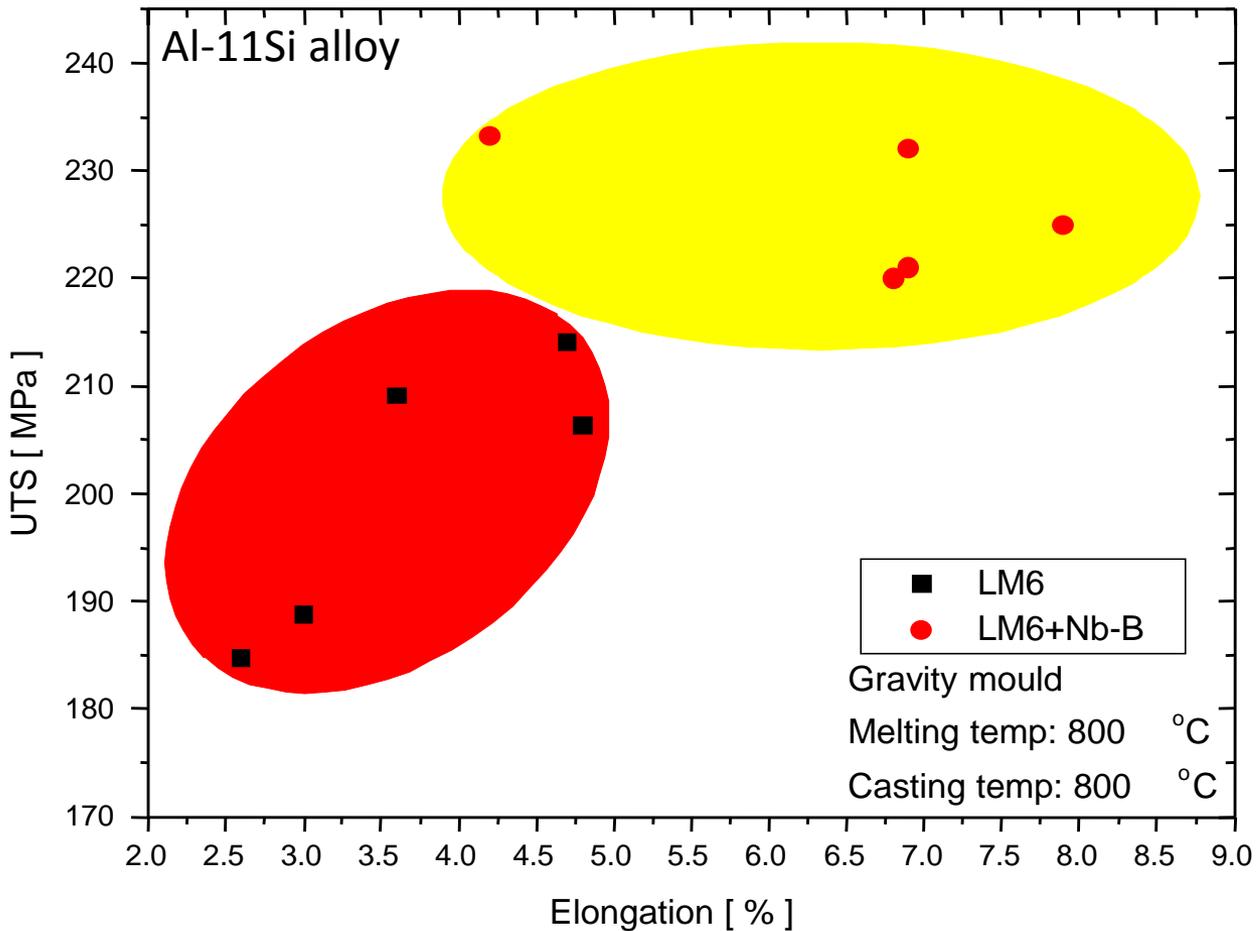
with Nb-B



# Nb-B Grain Refiner for Al-Si cast alloys

- Highly effective for Al-Si alloys & Mg alloys
- Fine & uniform grain structure
- Grain size is less sensitive to cooling rate
- Highly effective in sand casting cooling conditions
- Reduced porosity & macro defects
- Fine eutectic structure & intermetallics
- Improved ductility & strength**
- Tolerant to Fe contamination
- Recycling of Al-Si scrap

# Improved strength & ductility



Machined from cast bars

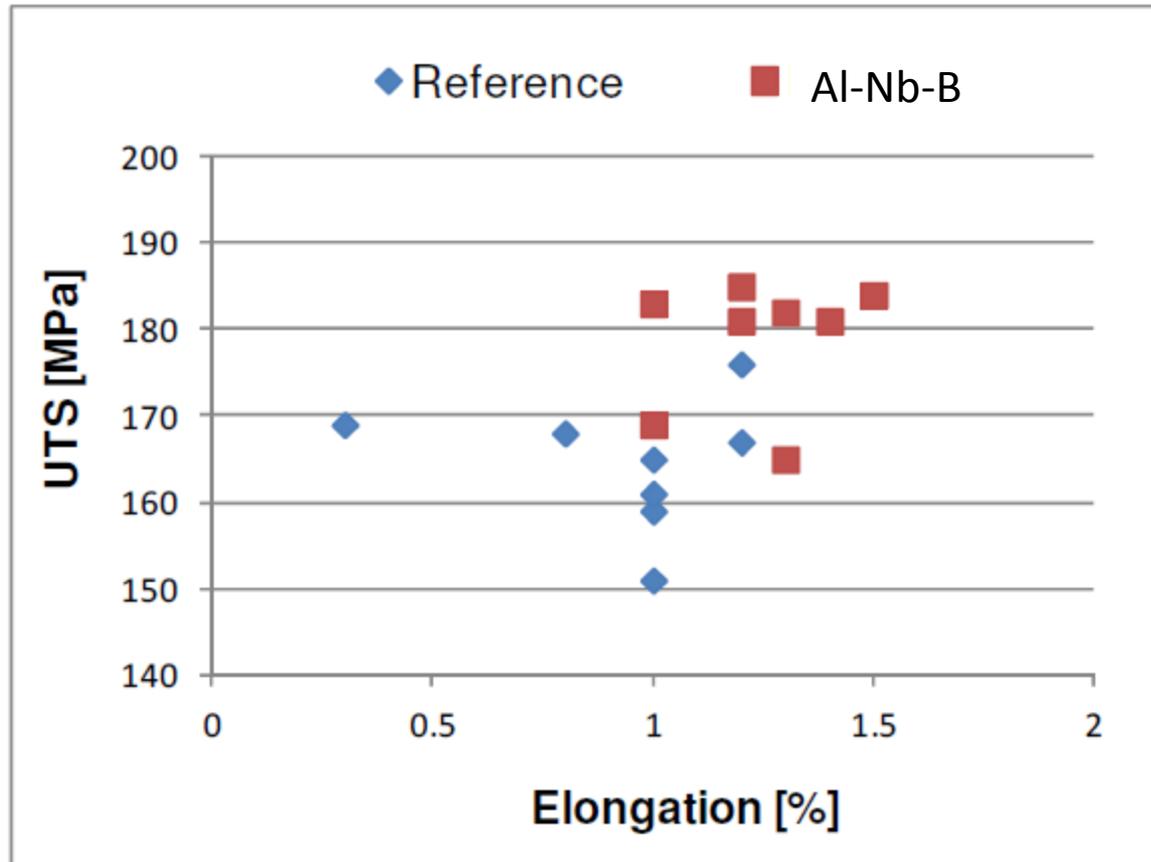
Improved

- Crash performance
- Fatigue performance

yield

82.4 Mpa → 102 Mpa

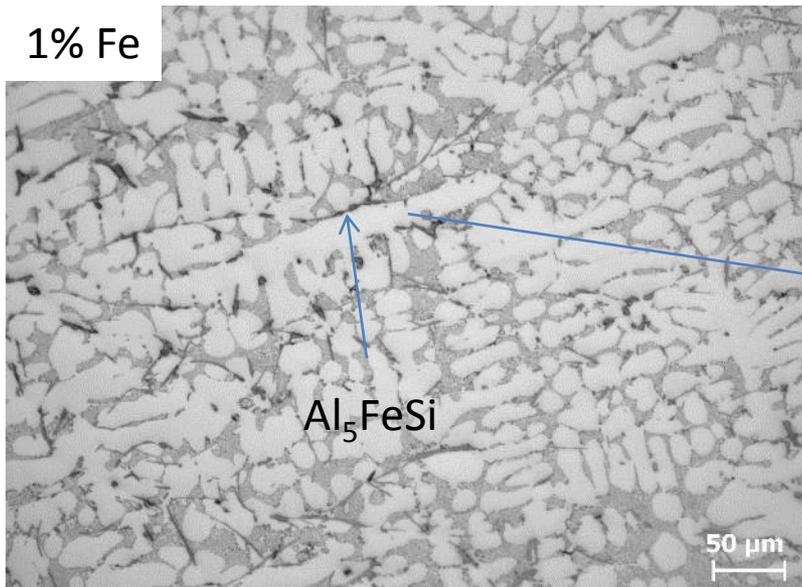
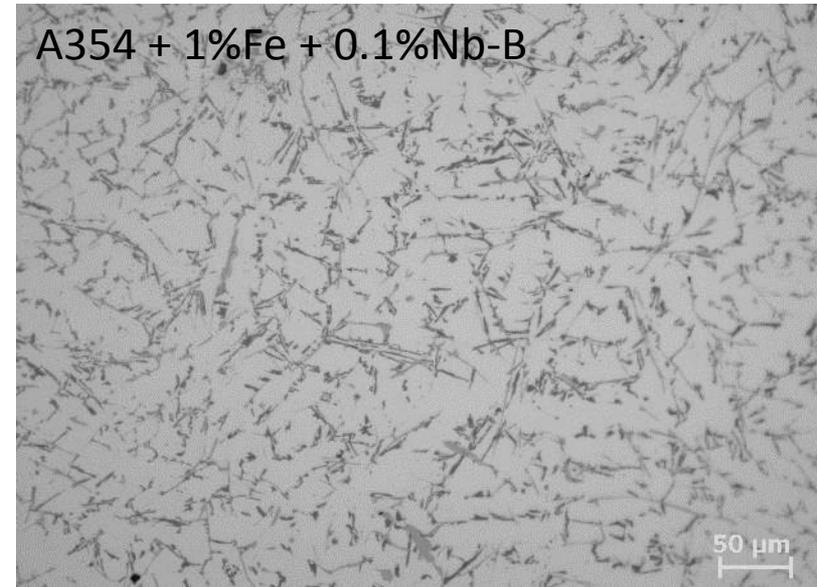
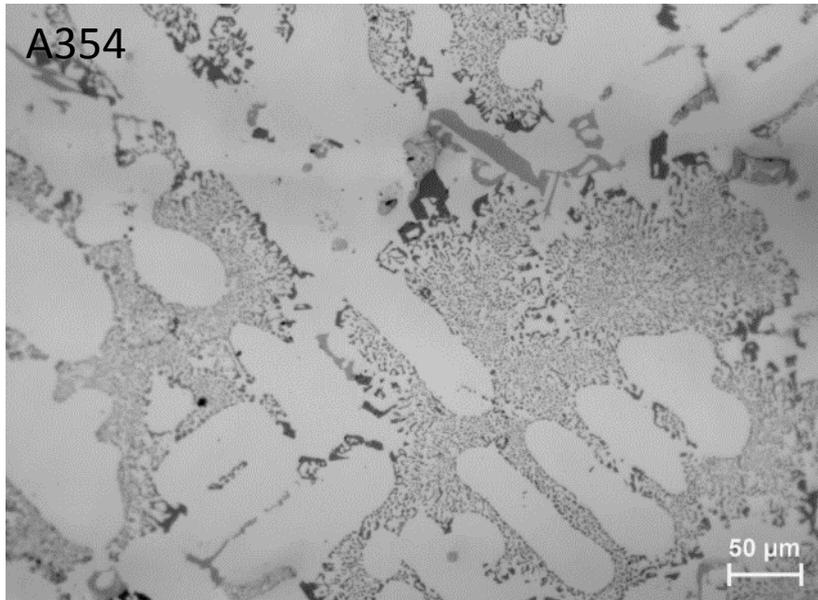
# Al-13Si piston alloy



# Nb-B Grain Refiner for Al-Si cast alloys

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- ❑ Improved ductility & strength
- ❑ **Tolerant to Fe contamination**
- ❑ Recycling of Al-Si scrap

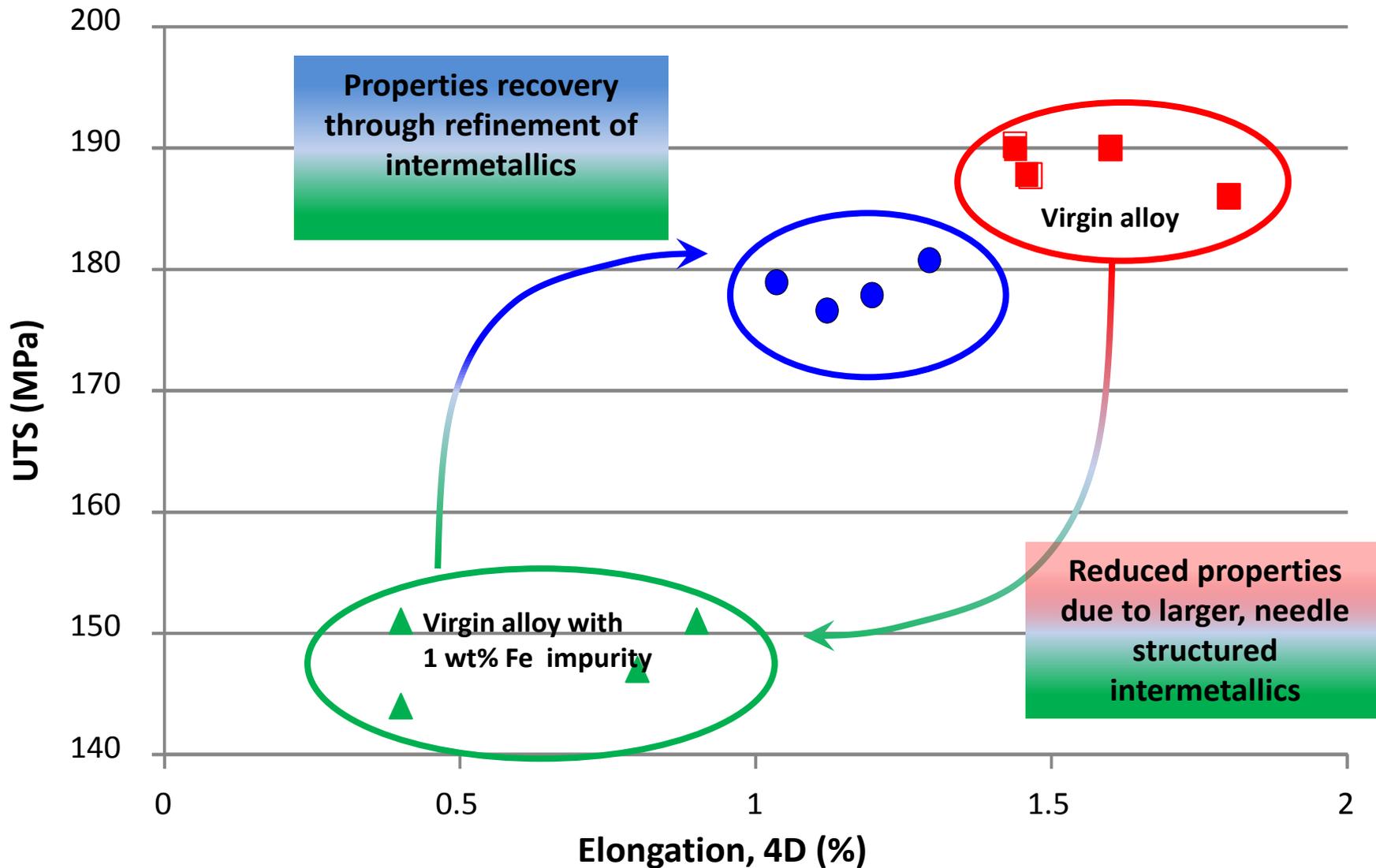
# Control of Fe-intermetallics in Al scrap



Grain refiner to control  
Al-Fe-Si intermetallics

Al-Fe-Si large needle structure  
detrimental to mechanical properties

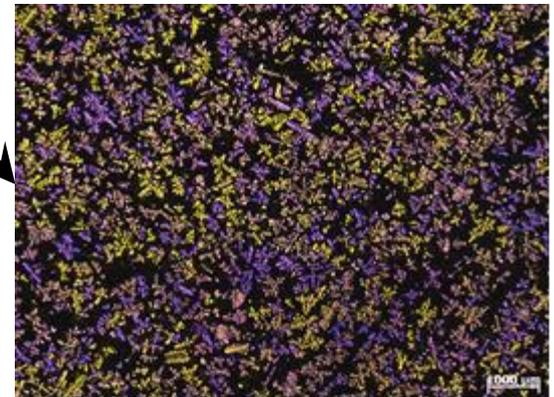
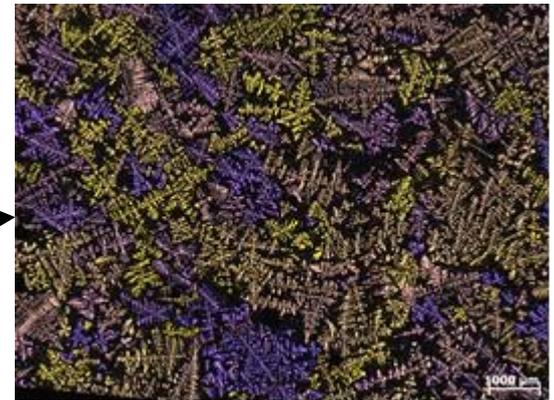
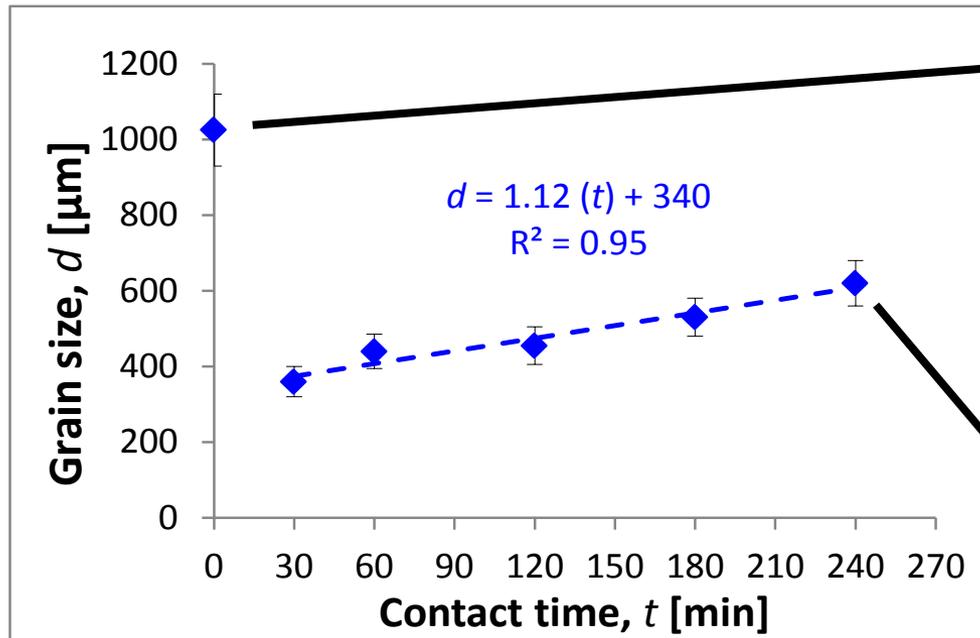
# Recovery of properties in Fe-rich aluminium scrap



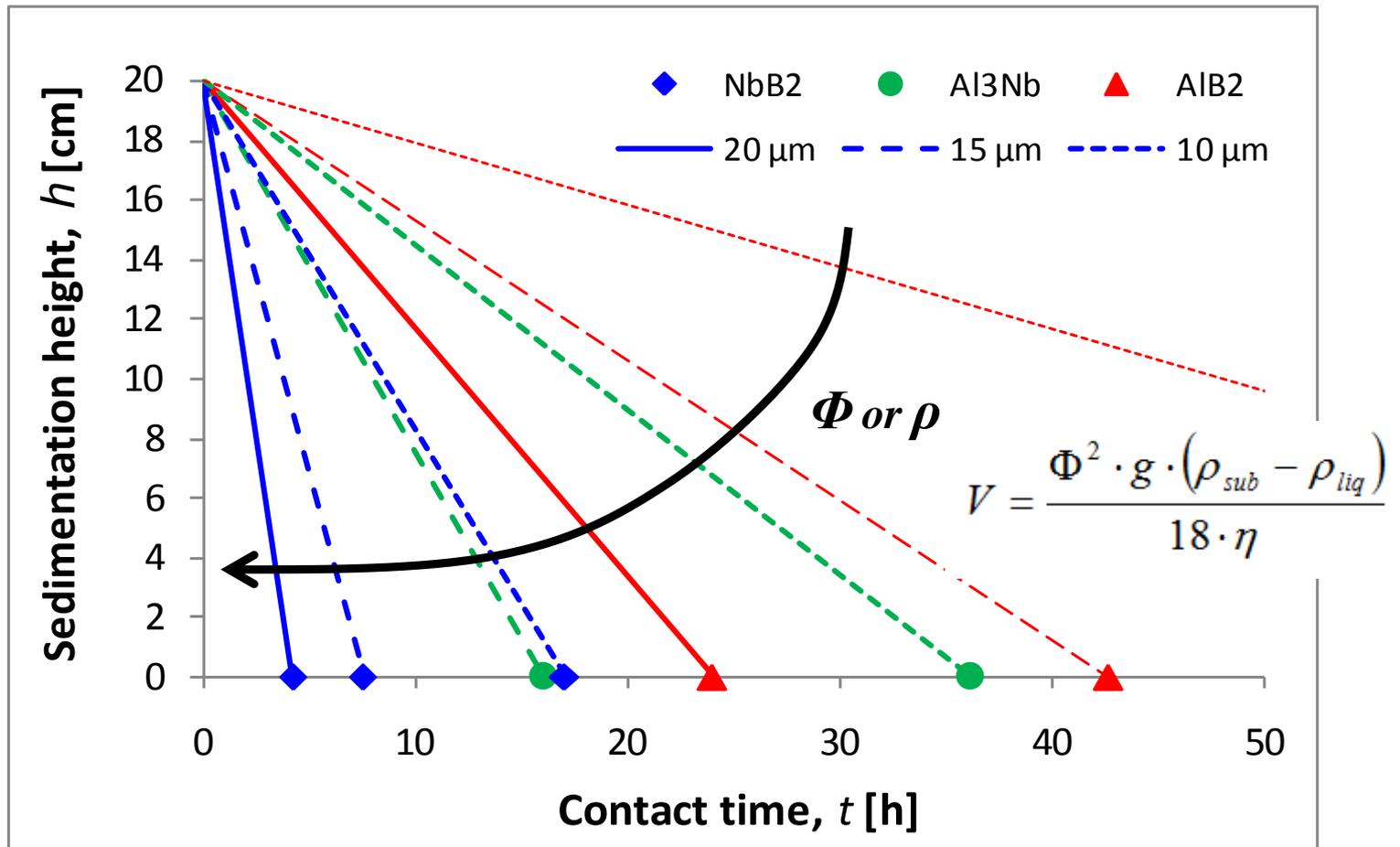
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- Improved ductility & strength
- Tolerant to Fe contamination
- Re-melting & Fading study**

# FADING STUDY

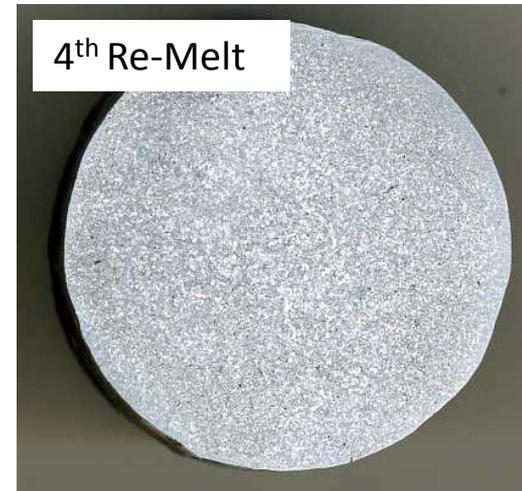
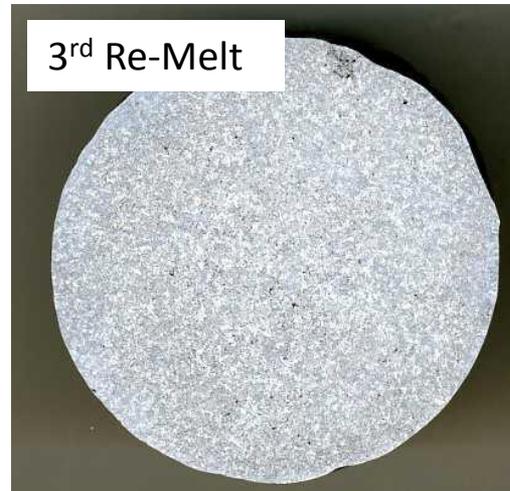
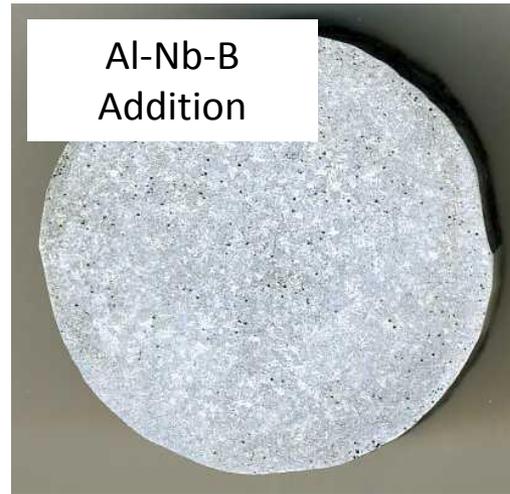


# REQUIRED TIME TO SEDIMENT/FADE



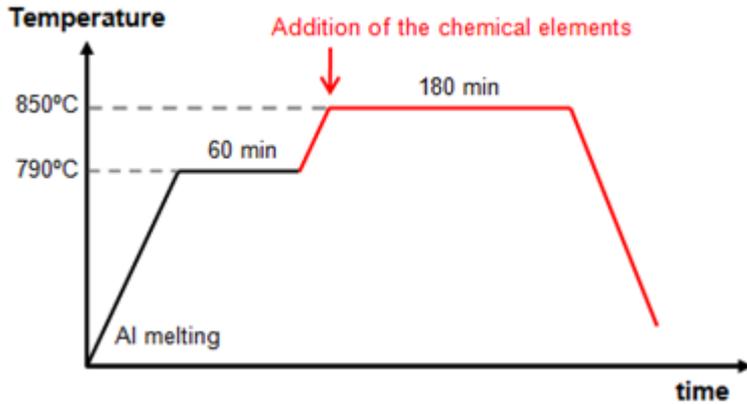
# Remelting

## Al-9Si-1.5Cu-0.6Mg-0.15Fe / RE-MELT



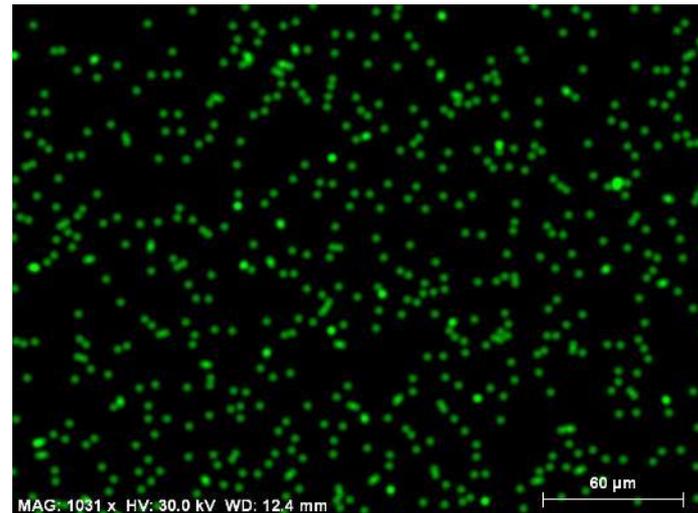
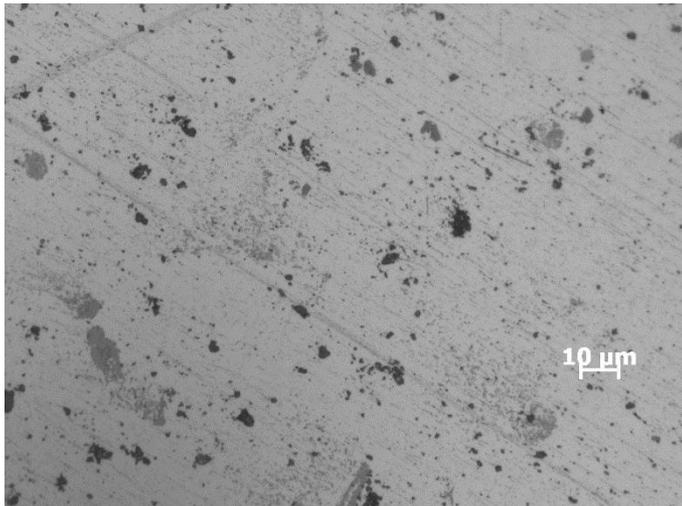
# **MASTER ALLOY DEVELOPMENT**

# Al-Nb-B Master Alloy

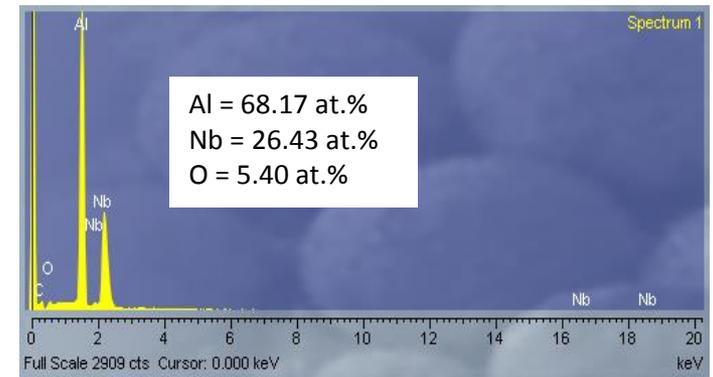
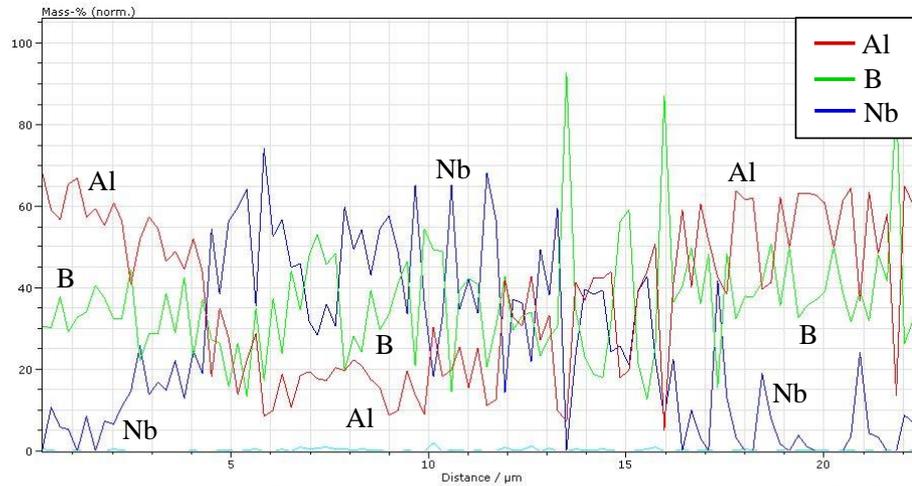
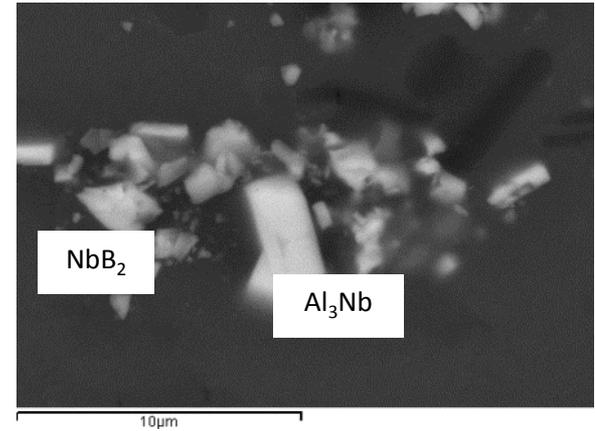
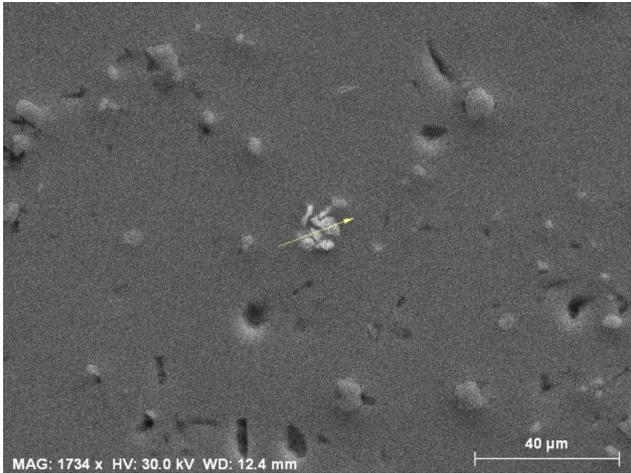


1. Nb metallic powder  
+ KBF<sub>4</sub>

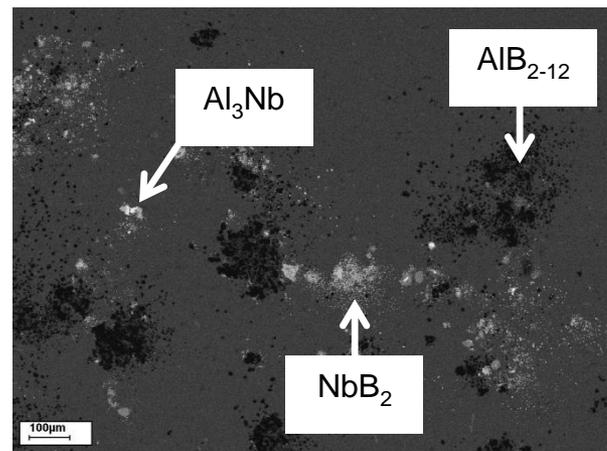
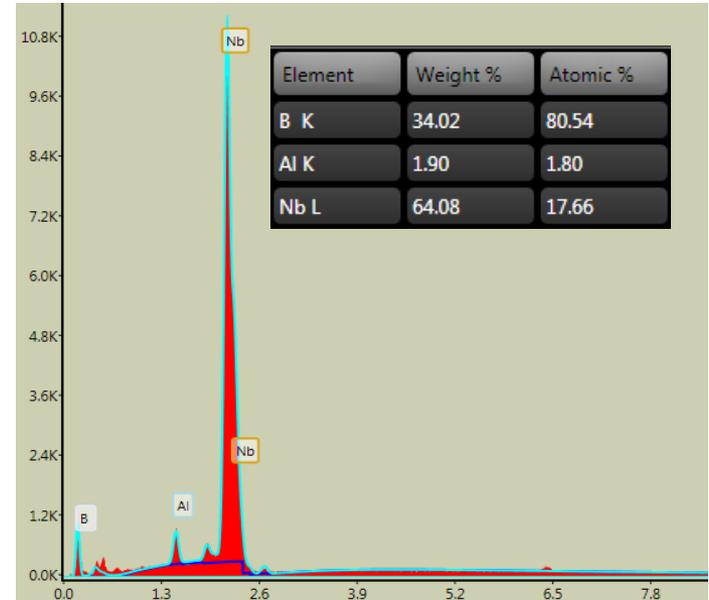
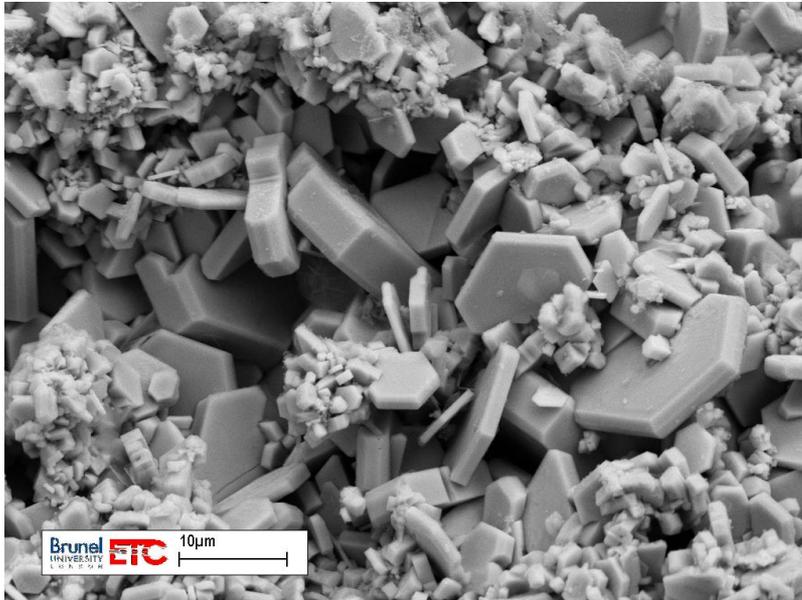
2. Addition of Nb metallic powder to diluted Al-B master alloy



# MASTER ALLOY (METHOD 1)

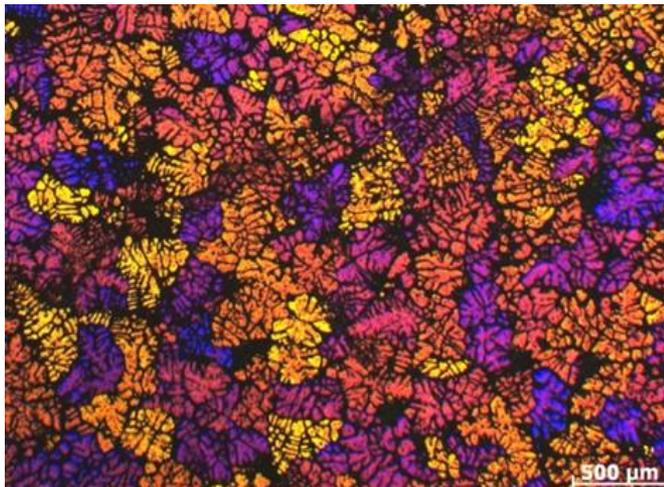
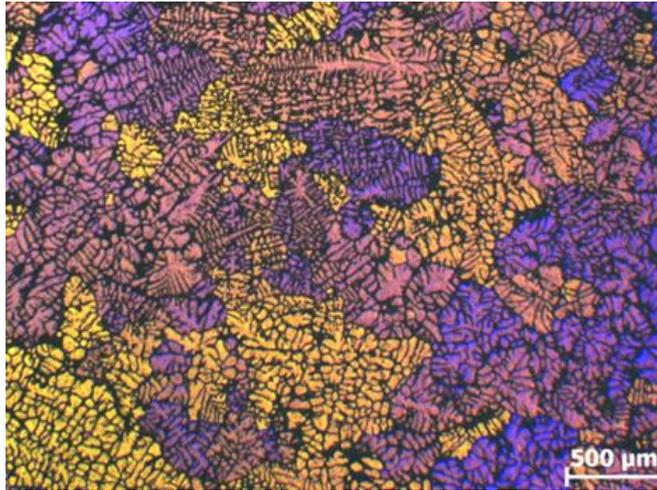


# Al-2Nb-2B (METHOD 2)

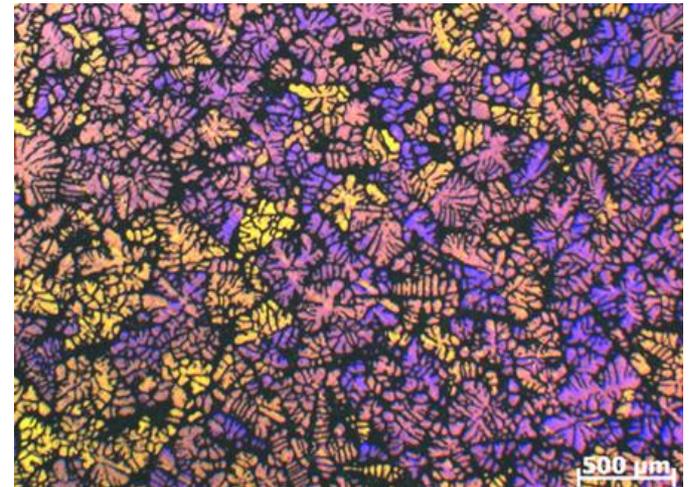


# Al-4Nb-1B on LM25

Reference

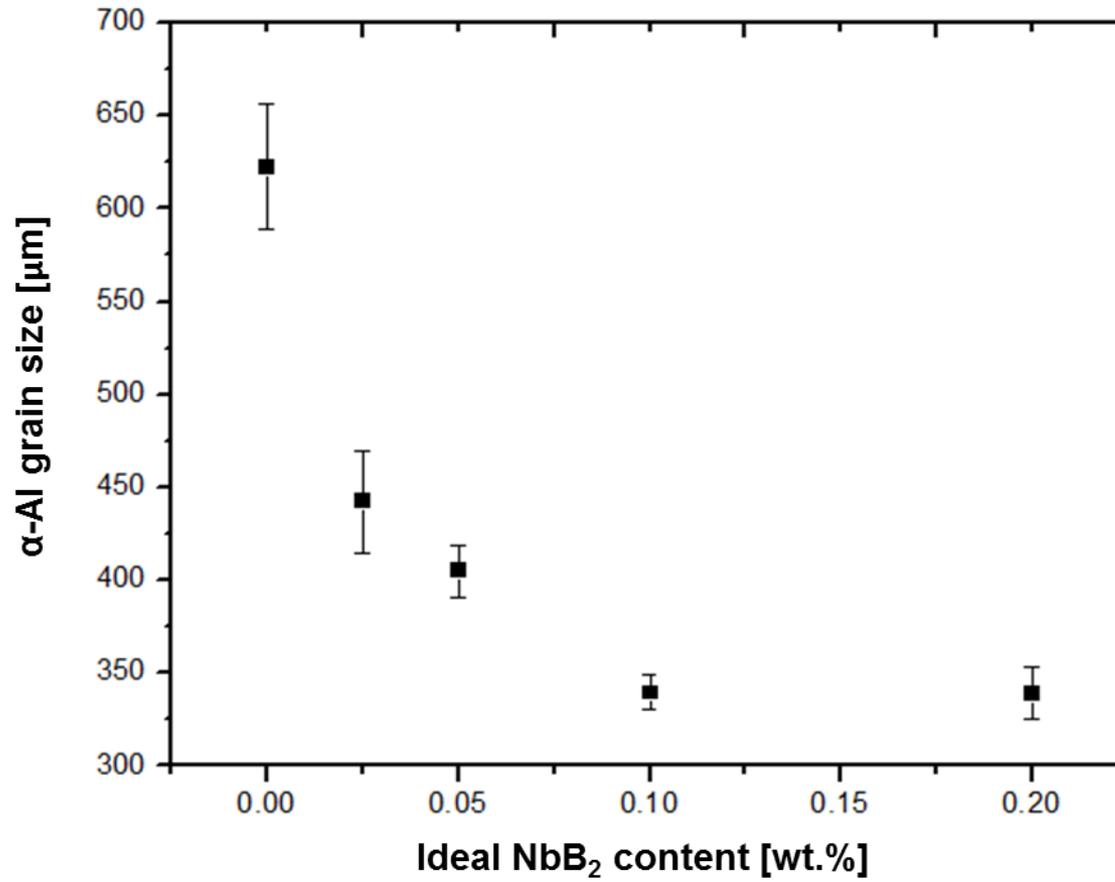


Nb-B (M.A.)

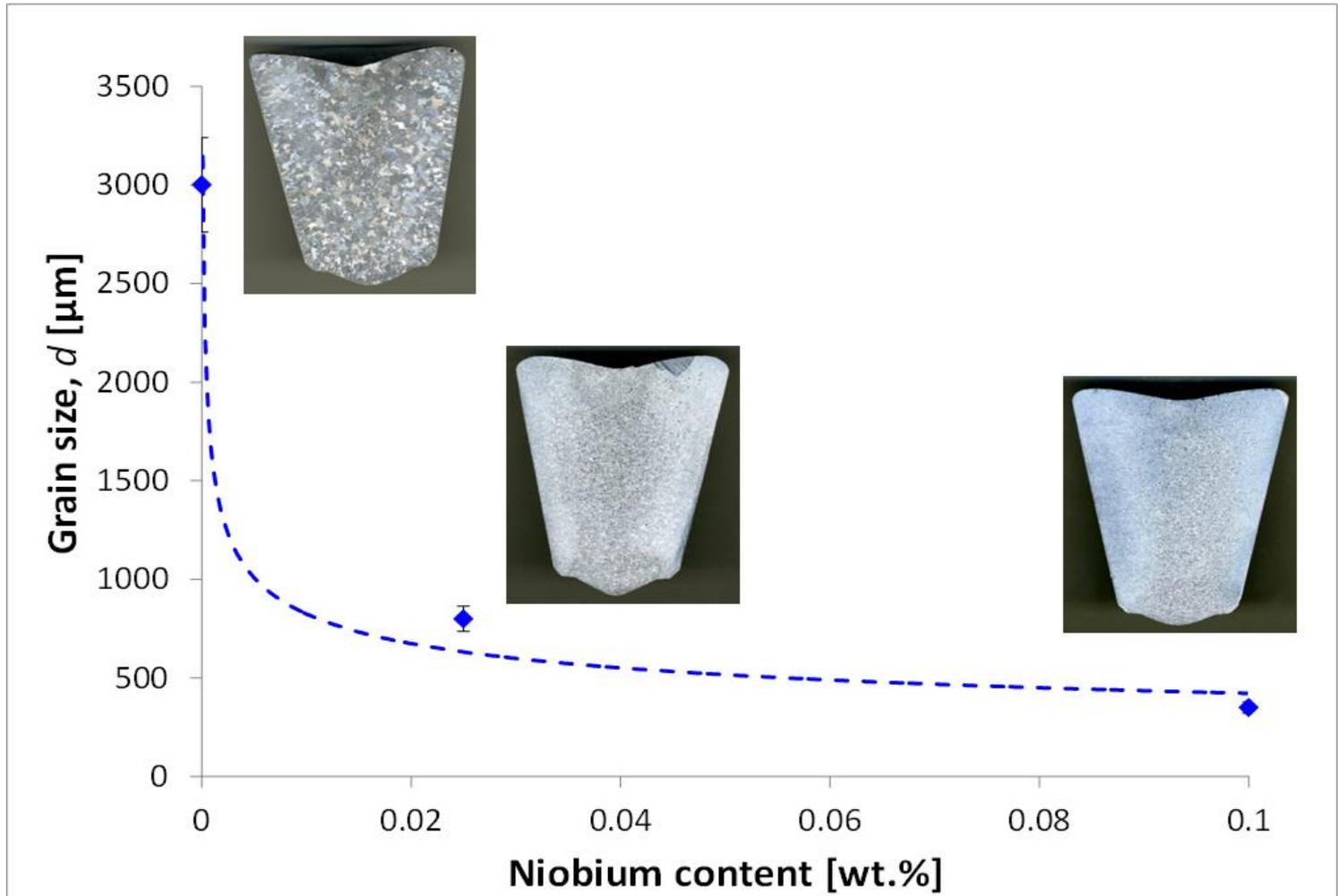


Al-Nb-B (powders)

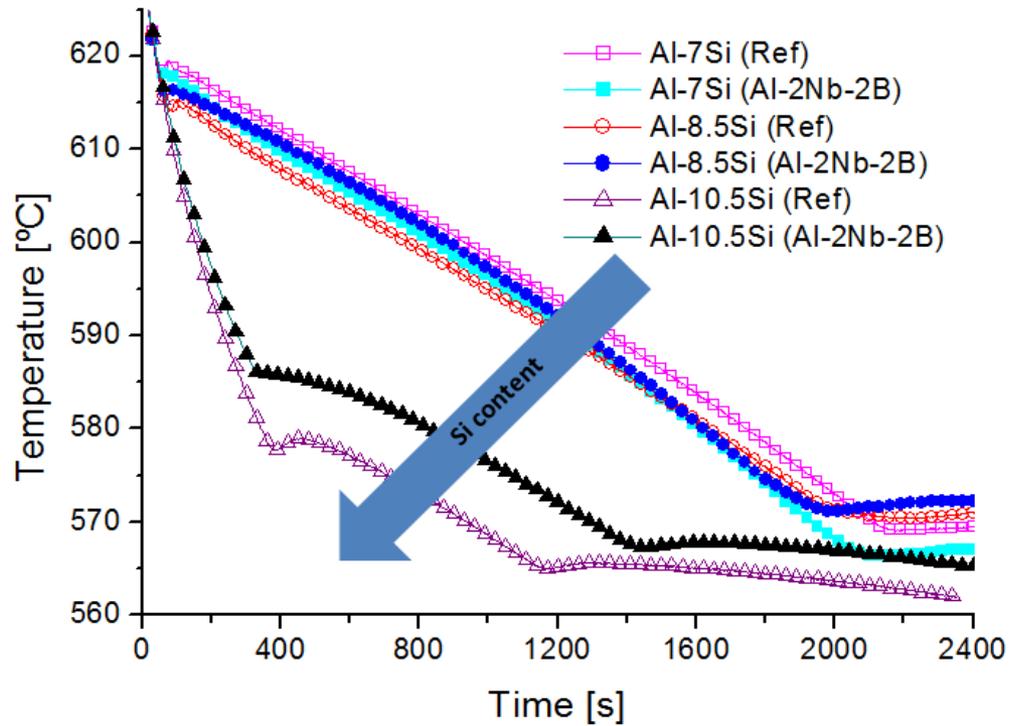
## Al-4Nb-1B ON LM6



## Al-2Nb-1B ON Al-10Si

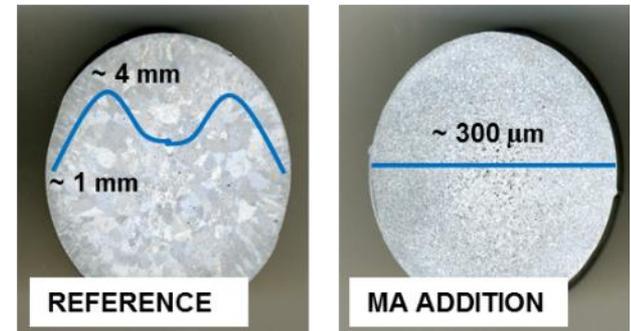
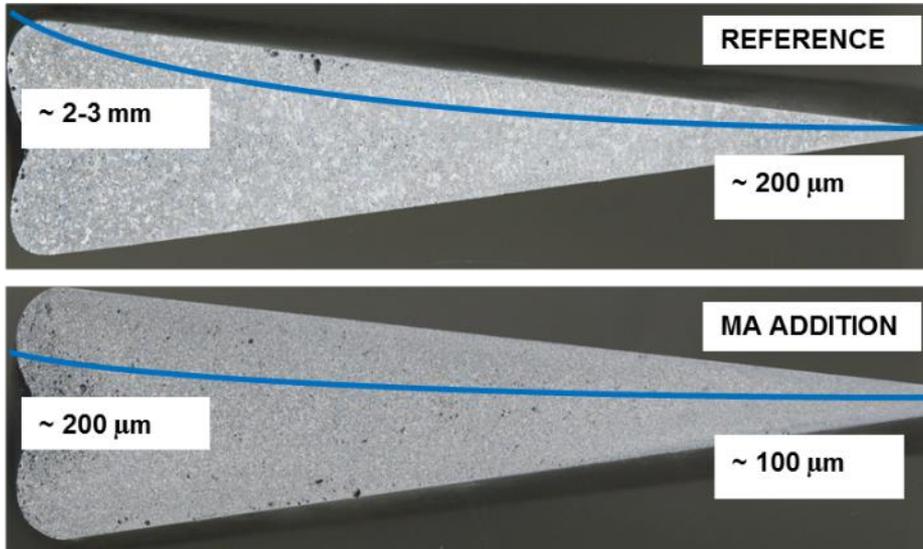


# EFFECT OF Al-2Nb-2B ON UNDERCOOLING FOR COMMERCIAL Al-Si ALLOYS

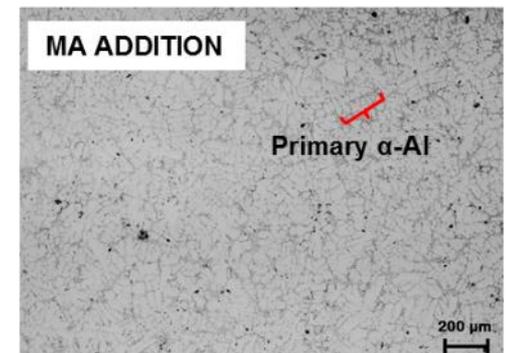
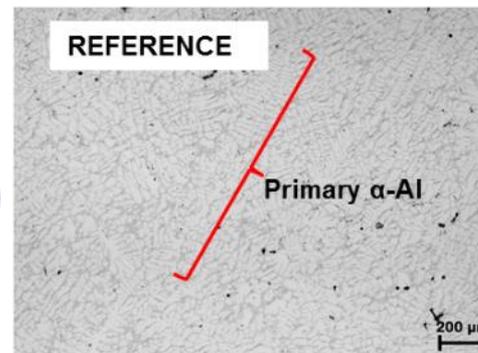


Material		Si content [wt. %]		
		7	8.5	10.5
<i>Reference</i>	$\Delta T_{\alpha}$	1.7	1.4	1.4
	$\Delta T_{eu}$	1.4	1.8	0.9
<i>Al-2Nb-2B master alloy addition</i>	$\Delta T_{\alpha}$	0.6	0.3	0.4
	$\Delta T_{eu}$	1.1	1.3	0.7

# EFFECT OF Al-2Nb-2B ON COMMERCIAL Al-Si ALLOYS



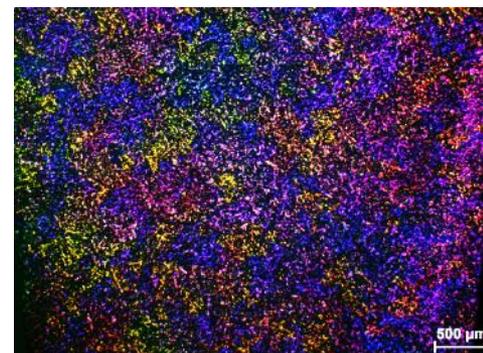
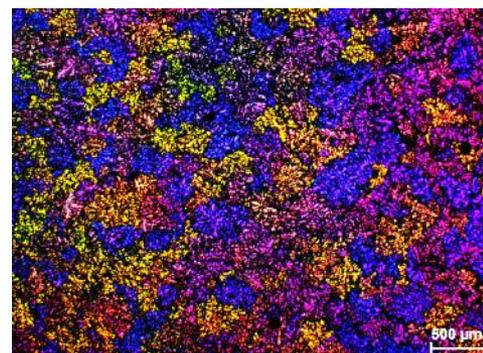
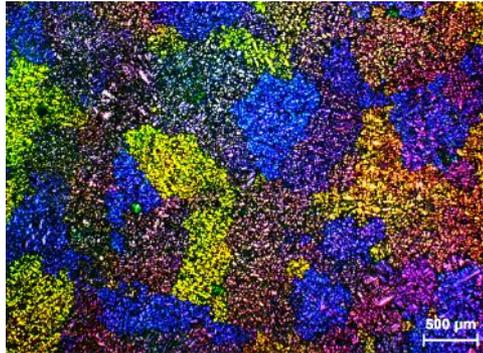
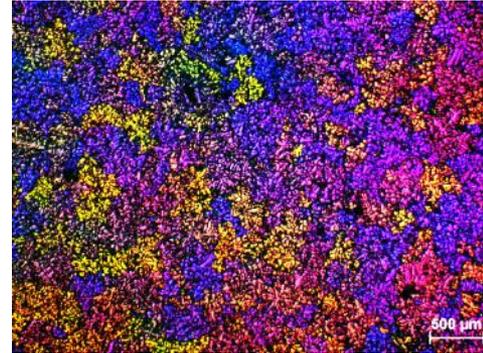
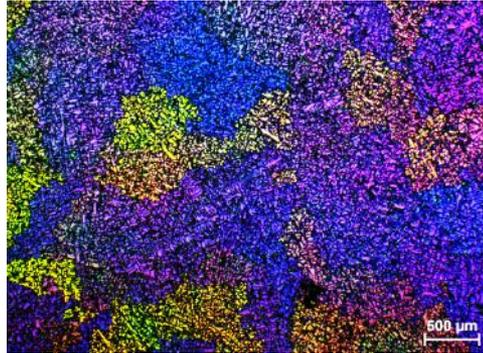
➤ **HYPO-EUTECTIC  
ALLOYS (LM24/25)**



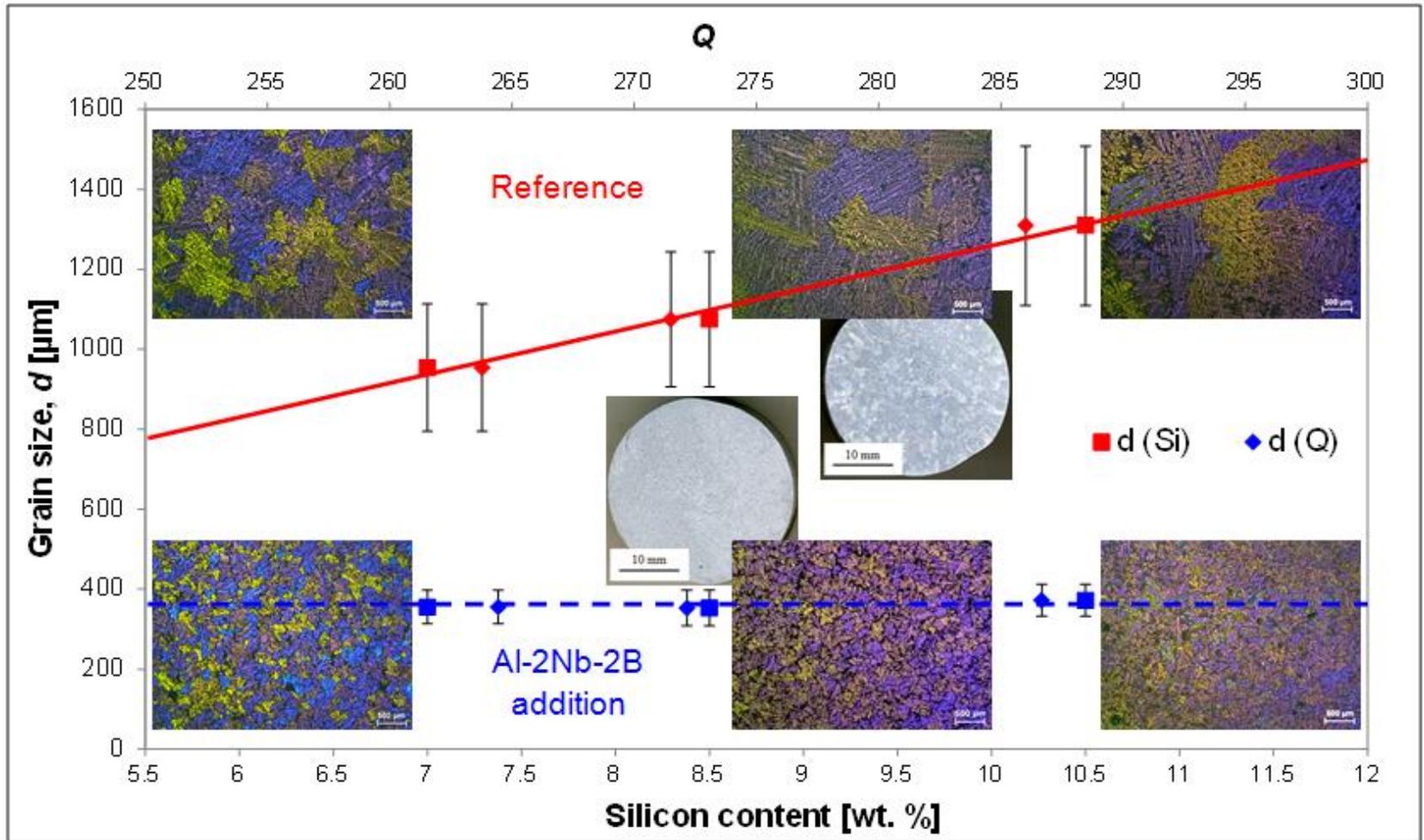
# EFFECT OF Al-2Nb-2B ON COMMERCIAL Al-Si ALLOYS

PISTON ALLOY (HYPER-EUTECTIC: 13 wt.% Si)

740°C

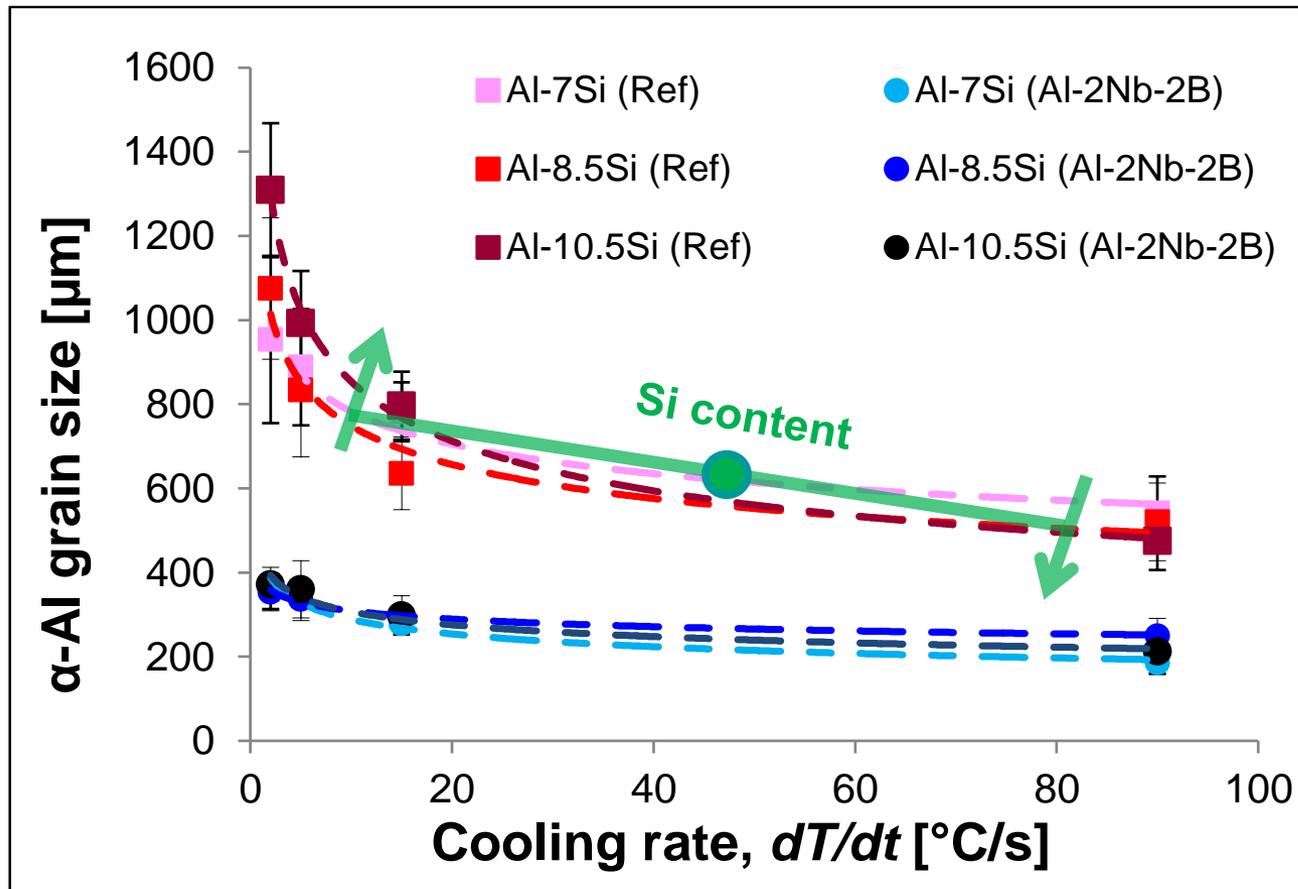


# EFFECT OF Al-2Nb-2B ON COMMERCIAL Al-Si ALLOYS

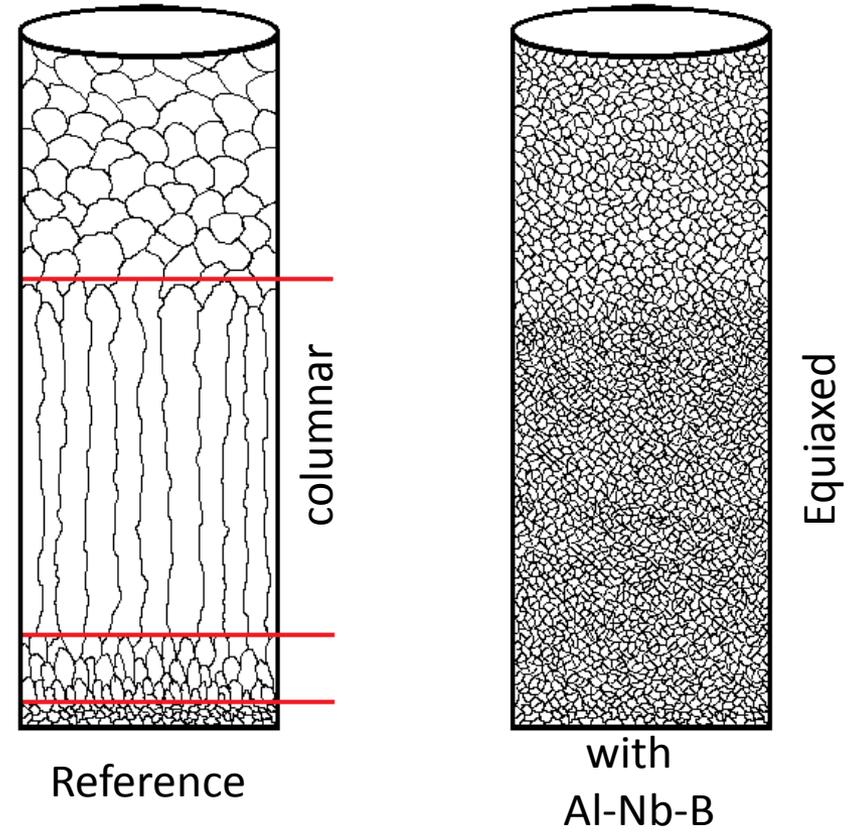


740 °C

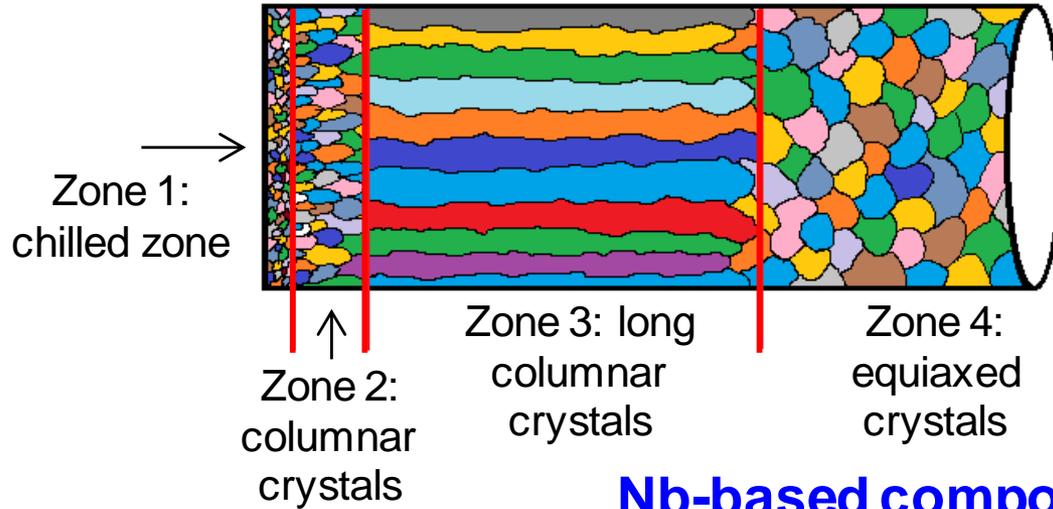
# EFFECT OF Al-2Nb-2B ON COMMERCIAL Al-Si ALLOYS



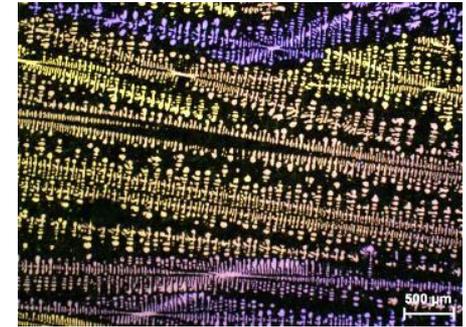
# Al-10 Si alloy - Direct Chill Cast Billets



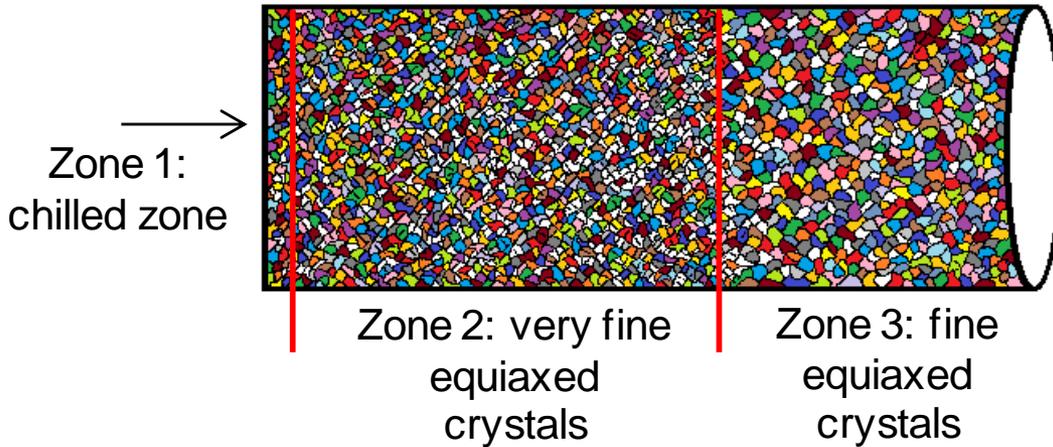
# Al-10Si DC billets



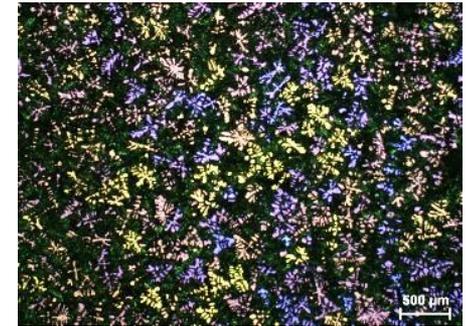
Reference

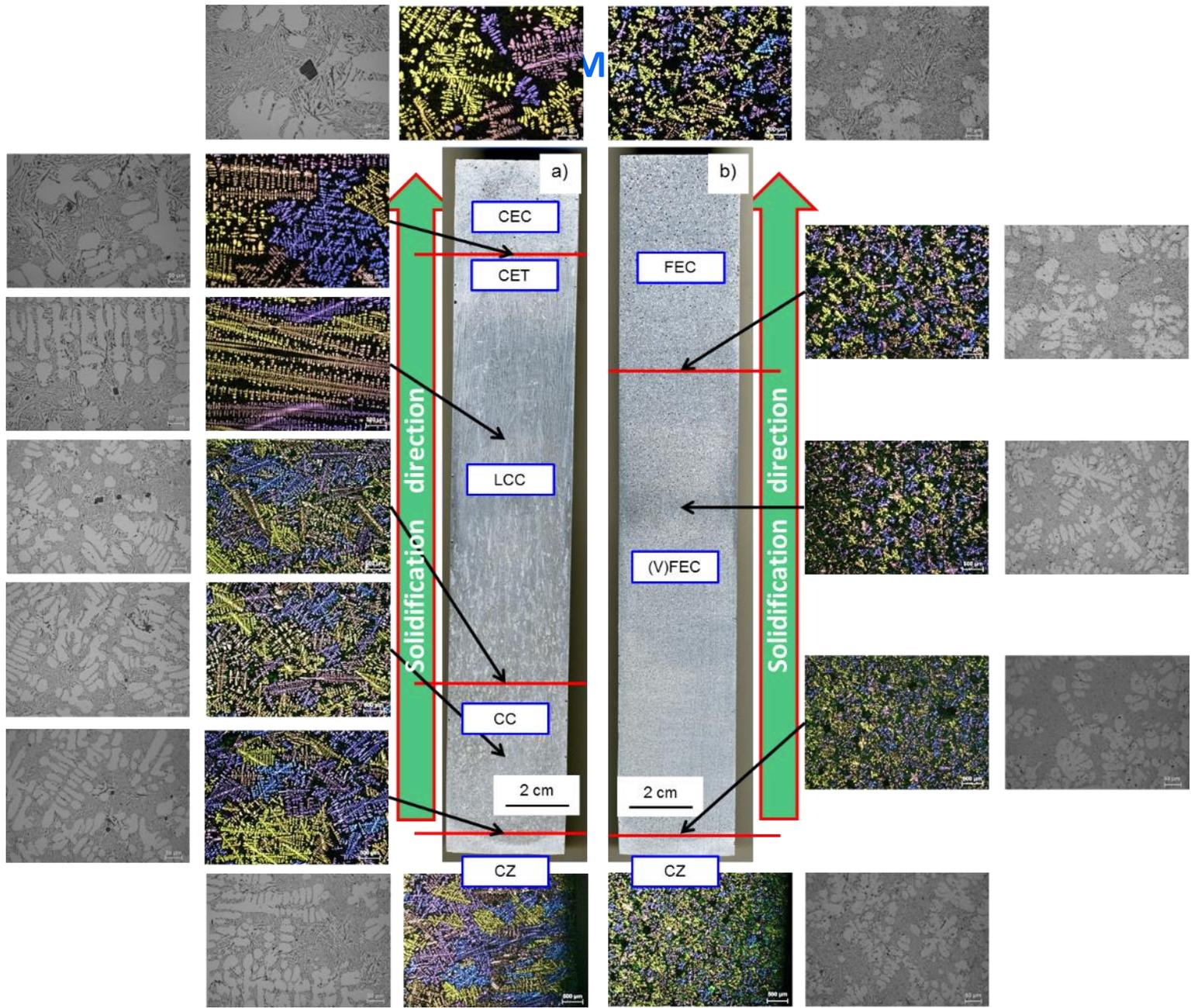


Nb-based compounds as heterogeneous nuclei



Inoculated





**Comparative study between Al-Nb-B and Al-5Ti-B master alloys**

# Comparison between Ti-B *and* Nb-B

Al-10Si

~ 4-5 mm

Al-2Nb-B master alloy addition

~ 300  $\mu\text{m}$

Al-5Ti-B master alloy addition

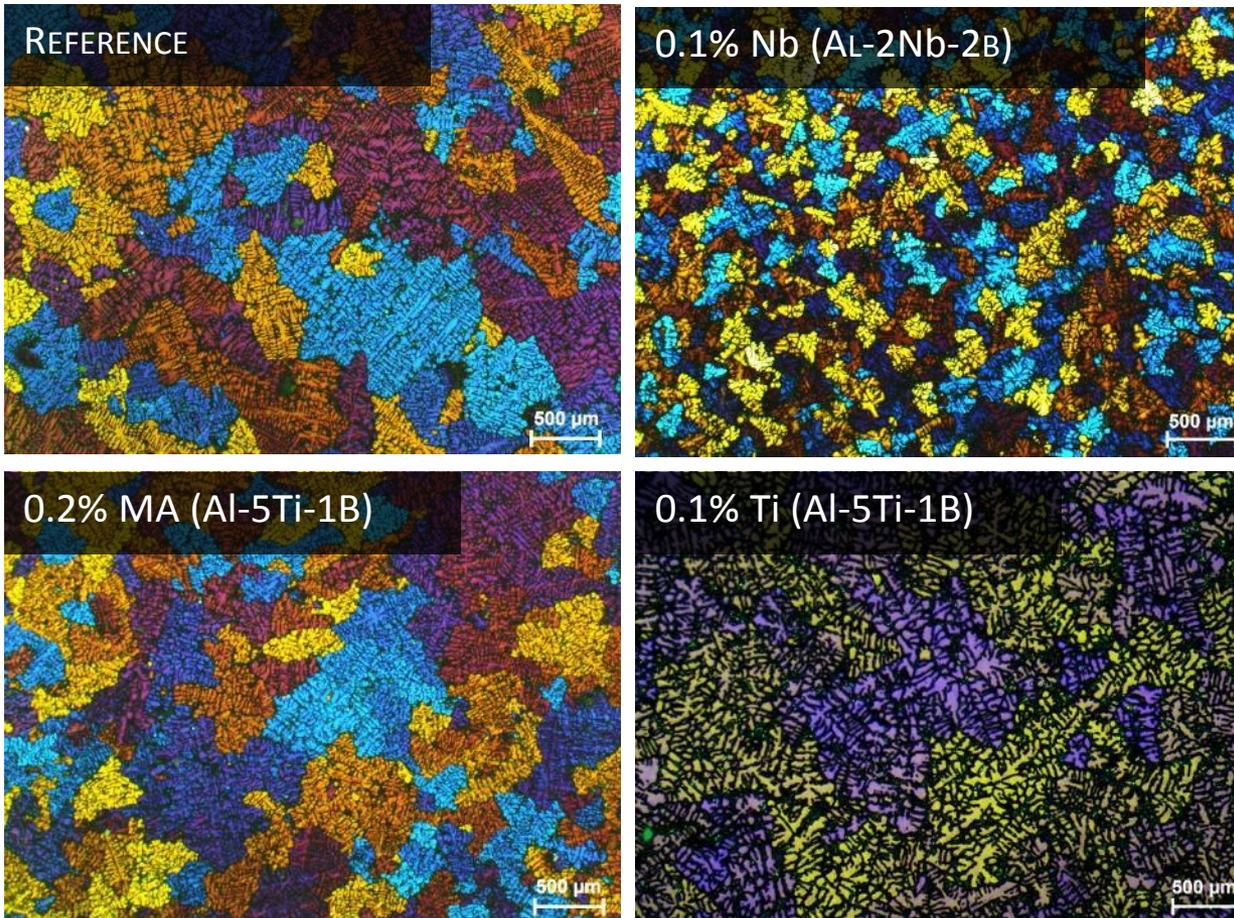
0.1% Nb

0.1%Ti

# GRAIN REFINEMENT EFFICIENCY COMPARISON

WHEEL ALLOYS (HYPO-EUTECTIC: 7 wt.% Si)

740°C



# Summary

- Nb-B addition to Al-Si melt refines the grain structure of casting
- End-user benefits:
  - Improved strength & ductility
    - ✓ Lighter/thinner structures
  - Homogeneous properties (thick & thin sections)
    - ✓ Complex structures
  - Tolerant to Fe contamination
    - ✓ Closed loop recycling of scrap containing higher Fe
  - Reduced shrinkage porosity - Improved soundness
    - ✓ Component rejection ratio can be minimised