Grain size control in steel by means of dispersed non-metallic inclusions GRAINCONT

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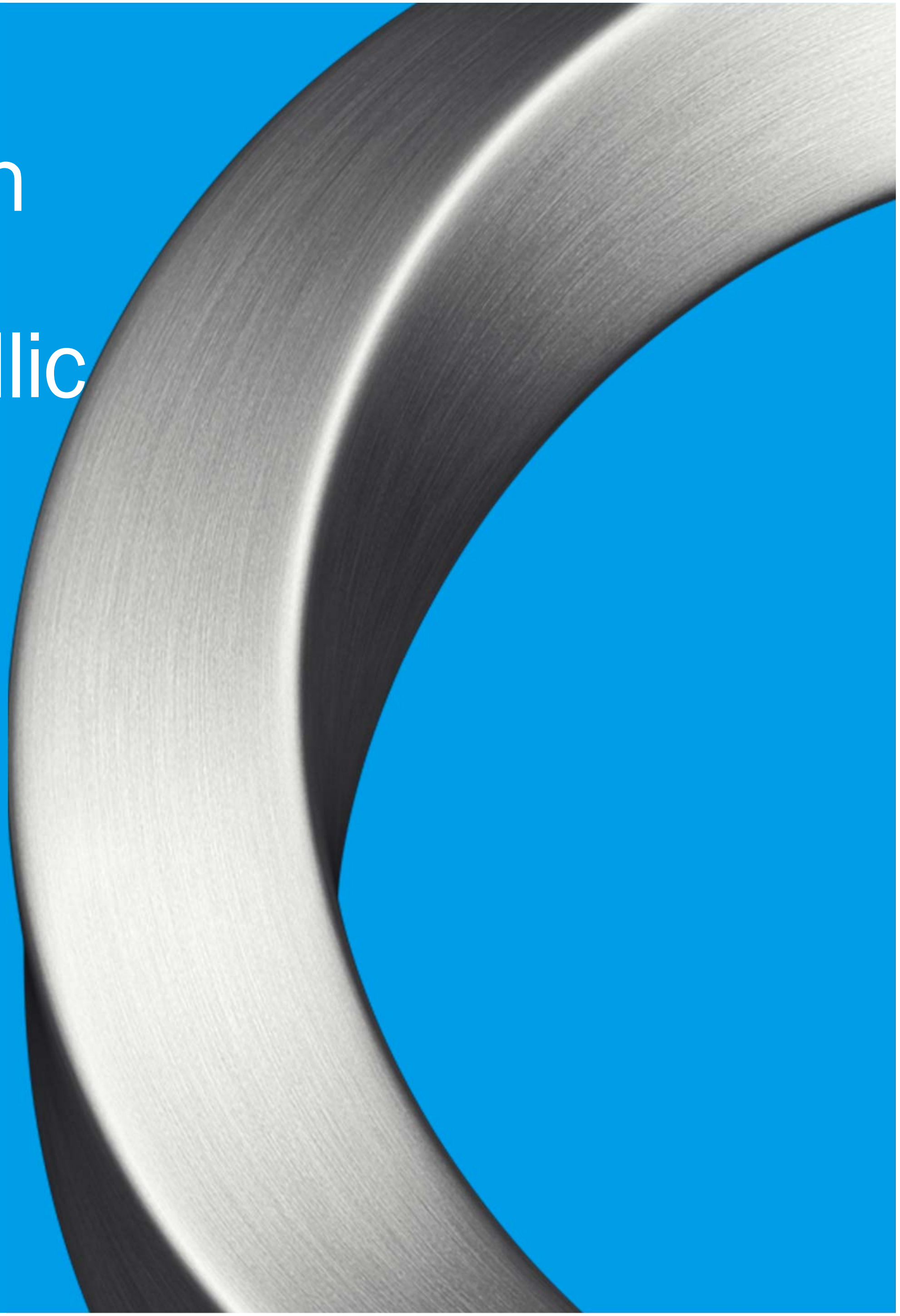
2017-05-22



GRAINCONT

The overall project goal was to create a ground for an industrial technology utilising the concept of grain size control by means of dispersoidic inoculants placed into the steel microstructure.

This was to be achieved by:

- Create and improve the basic knowledge of grain refining alloys with dispersed non-metallic inclusions (e.g. particles)
- Develop and establish a new addition technique applicable for steel continuous casting.

Partners

KTH

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TKK

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CRM

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ArcelorMittal

E. de Courcy, L. Chapuis, T. Iung

CdC

S. Ekerot

SINTEF

C. van der Eijk

WP-planning

- WP1: **Potential inclusions/particles**

To find best potential inclusions for grain refinement and to expand the basic knowledge of the thermodynamic equilibria and kinetic phenomena for establishing the necessary dispersion of non-metallic inclusions appropriate to act as inoculants.

- WP2: **System with steel**

To optimise the inoculants systems for different steel grades and to produce samples for further working and testing.

- WP3: **Produce inclusions**

To produce inoculants systems for further testing, but also as a demonstration of possible full scale production. The process routes to be investigated were (1) inoculant dispersoids in specially designed ferroalloys and (2) deoxidants systems able to be introduced prior to the casting.

- WP4: **Test addition**

To test and demonstrate ways of adding the inoculants systems to the molten steel just prior to the casting.

- WP5: **Evaluation of effect**

To evaluate the grain refinement effects on final properties of selected steel grades from tests being done in WP2 and WP4.

- WP6: **Final evaluation**

To evaluate the grain refinement effects and the production technologies developed in the project.

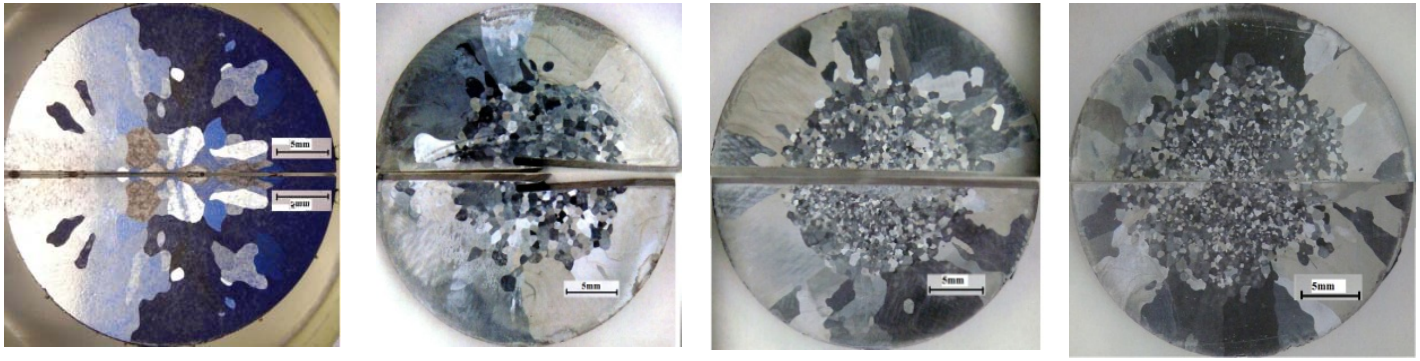
Thesis

Ph.D Defense 2010-05-20

outokumpu

“Inclusions and/or Particles Engineering for Grain Refining Purposes in an Fe-20mass% Cr Alloy”

Jesper Janis



Outline

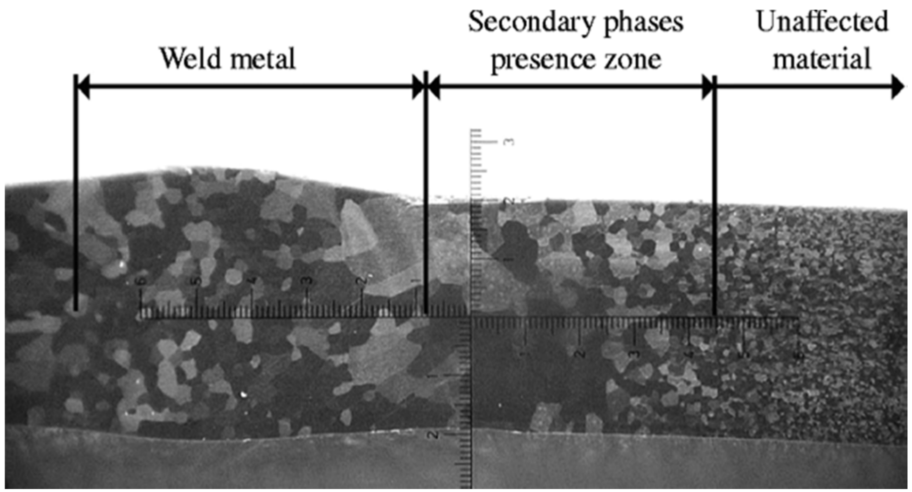
- Introduction - Grain refining of ferritic stainless steels
  - ➢ Characteristics of ferritic stainless steels
  - ➢ Main objectives
- Experimental work
  - ➢ Deoxidation experiments
  - ➢ Particles analysis
  - ➢ Precipitation and nucleation
  - ➢ Grain analysis
  - ➢ Pinning effect
- Results and discussion
- Conclusions
- Industrial aspects

Grain refining of ferritic stainless steel

Characteristics of ferritic stainless steels

+ High Cr content – good corrosion properties
+ Low Ni content – more stable and much lower alloying costs

- Problems with large as-cast grains – poor mechanical properties
- Ridging and roping
- Grain growth during welding

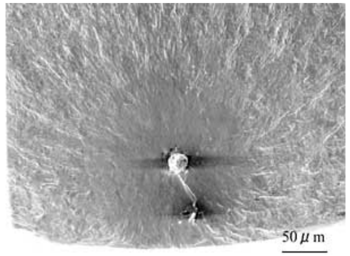


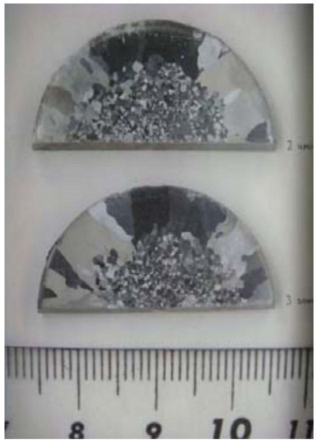
http://www.emeraldinsight.com/fig/1280560403009.png

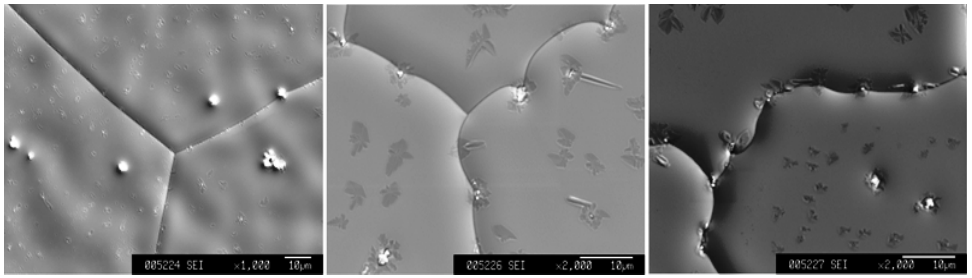
Inclusions or Particles

Inclusions: Bad reputation

- ✓Crack initiators
- ✓Pitting corrosion
- ✓Surface quality







Yield strength for a single crystal

Material constant

$$\sigma_s = \sigma_0 + \frac{k}{\sqrt{d}}$$

Yield strength

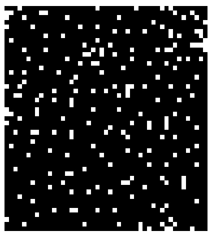
Grain diameter

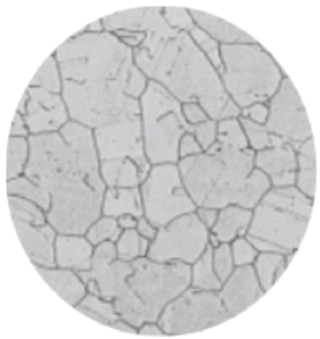
Particles: Good reputation

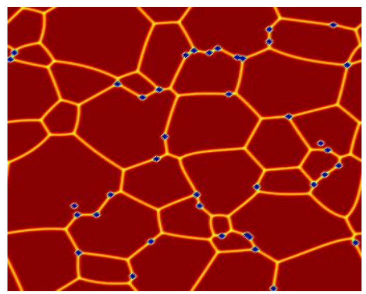
Main objectives

- Develop electrolytic extraction technique for high Cr alloys
- Create and study inclusions formation in a Fe-20mass% Cr alloy
- Investigate the grain refining possibilities of a Fe-20mass% Cr alloy by using fine inclusions (hereafter called particles)
- Observe and analyze the pinning effect of these particles

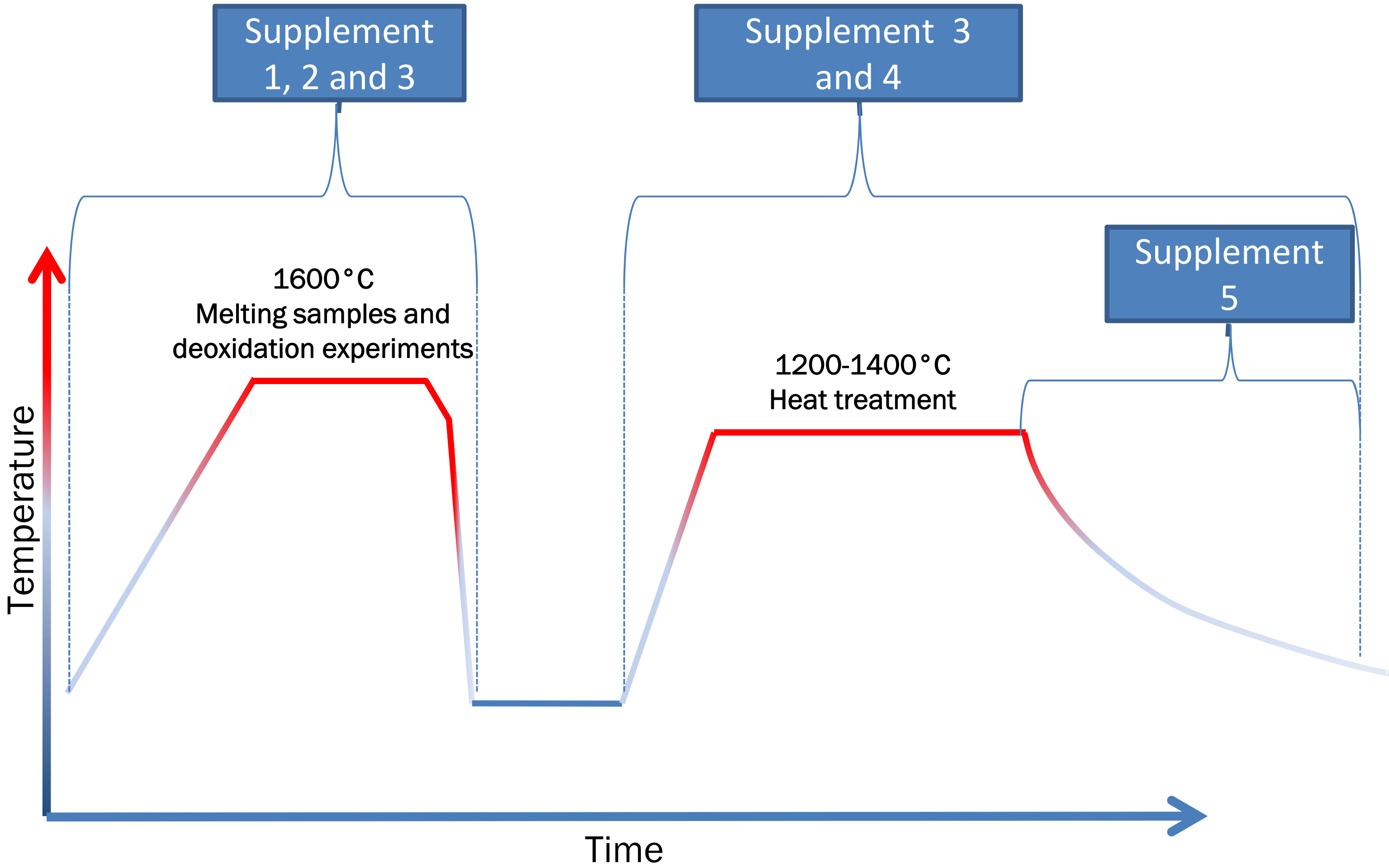
Create and study particles → Grain size determination with respect to particles → Grain pinning effect with respect to particles







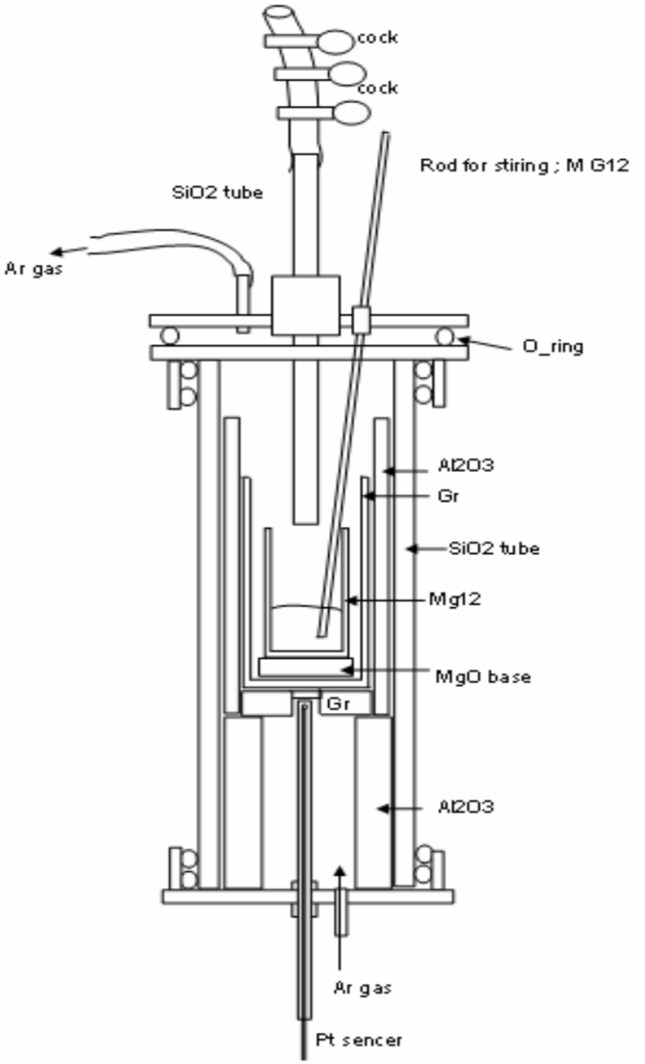
Connection of supplements

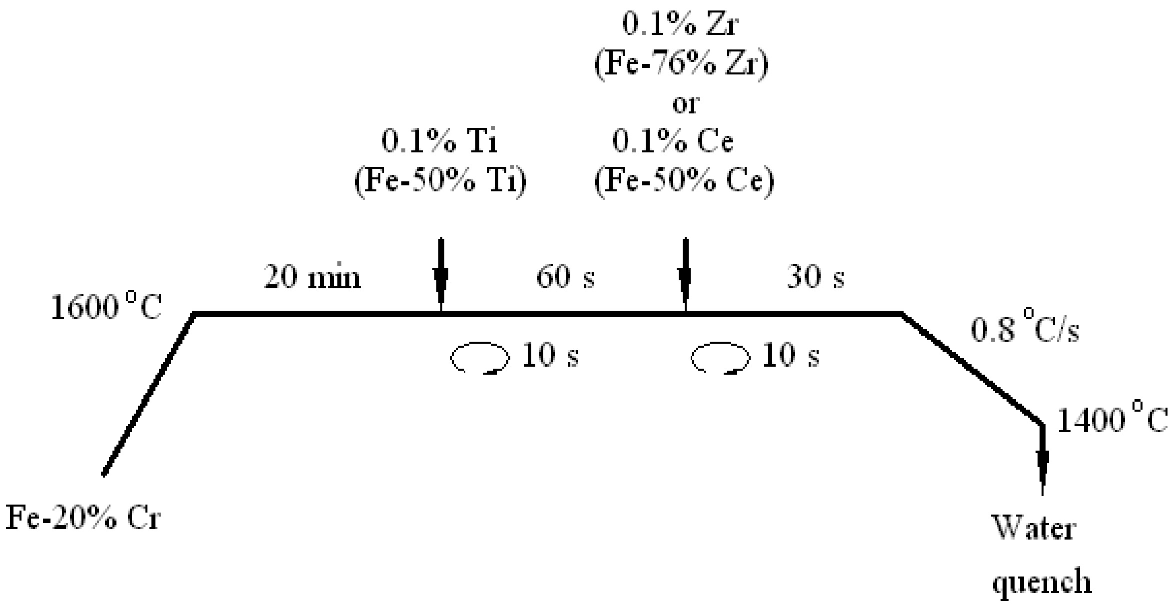


Experimental work

- Deoxidation experiments (High frequency induction furnace)
  - Making of samples with various addition Ti, Ce and Zr
  - Various N and O contents
- Electrolytic extraction
  - Particles analysis: size distribution, composition, morphology and number/mm^3
  - Analysis of soluble and insoluble elements
- Grain analysis (Image Analyzer)
  - Measurement of equiaxed grain size
- Thermodynamic calculations (Thermo-calc)
  - Precipitation phenomena
- In situ grain pinning analysis (CSLM)
  - Particles pinning effect
  - Shape of grain boundaries

Deoxidation experiments

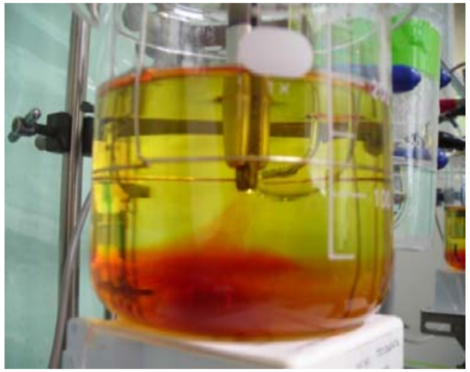


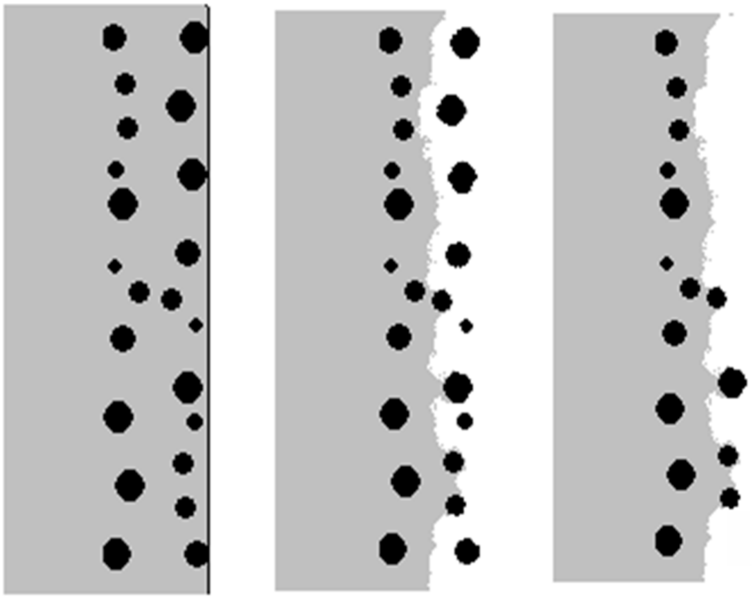


Particles analysis (Electrolytic extraction)

Electrolytic extraction

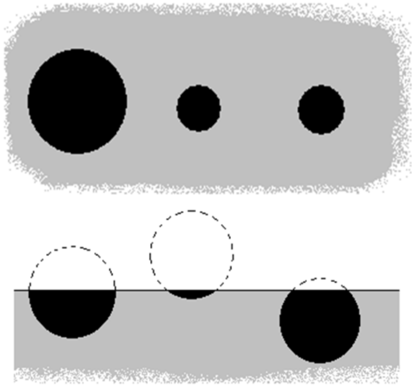
- Dissolves the matrix and releases the particles



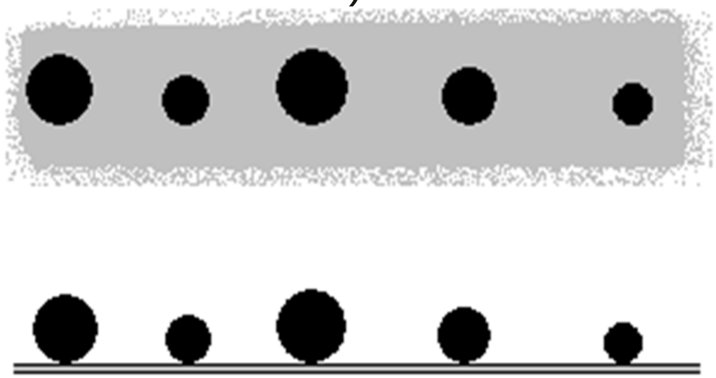


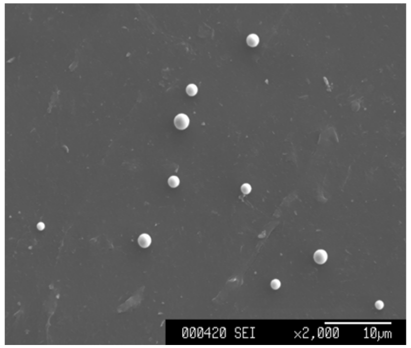
Observations in 2D vs. 3D

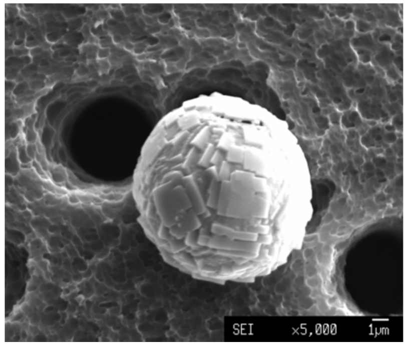
- 2D (Cross section)

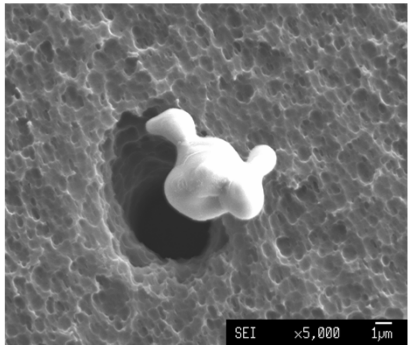


- 3D (Electrolytic extraction)







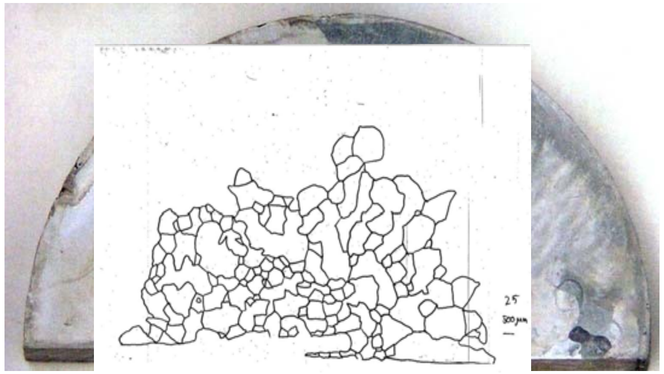


Thermodynamic calculations

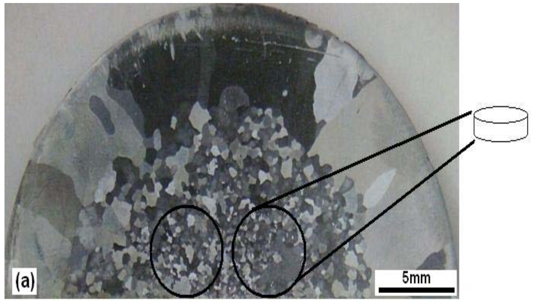
- Thermo-calc software
- Input experimental data from soluble and insoluble elements based on electrolytic extraction results
- Compare with analyzed precipitations
- Temperature vs. ppm of precipitated particles
- Show temperatures for particles precipitation
  - Primary precipitation
  - Secondary precipitation

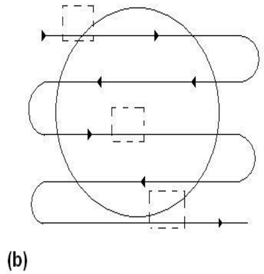
As-cast grain size analysis and pinning effect during heat treatment

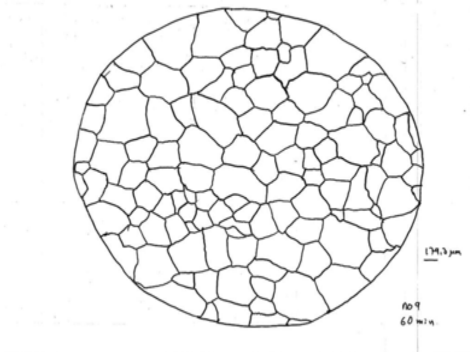
As-cast



Heat treatment



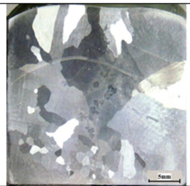
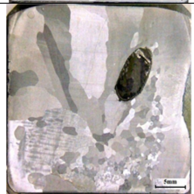
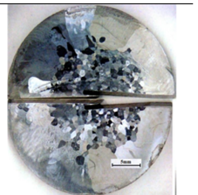
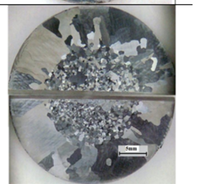
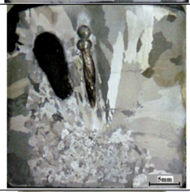
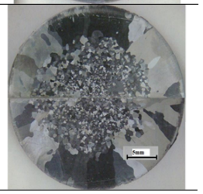
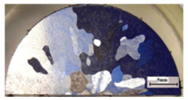
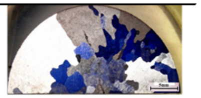




Confocal Scanning Laser Microscope

- Tohoku University
- In situ observations up to 1700°C
- Protected atmosphere
- Suitable for pinning effect observations
- Video recording

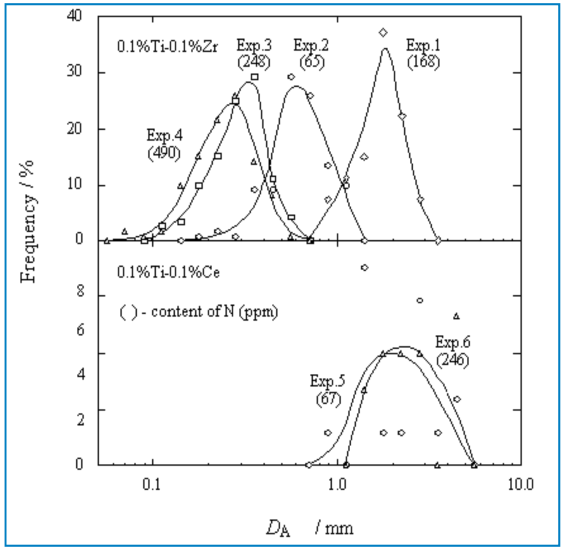
Grain refining results

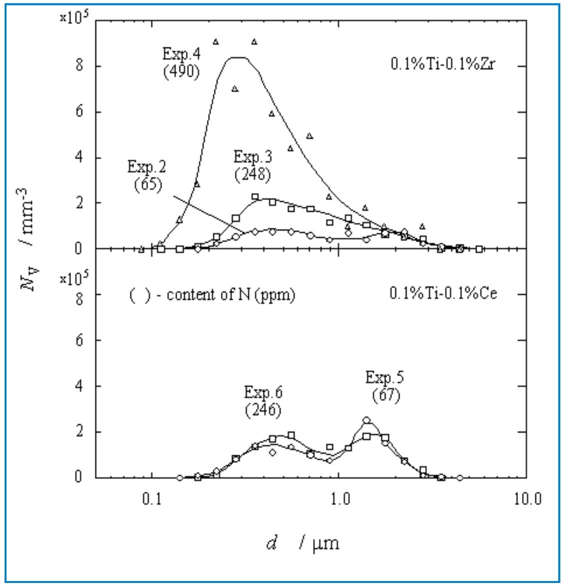
Exp No.	Deoxidation	Parameters	Vertical slice	Horizontal slices
1	-	[T.O] = 250 ppm [T.N] = 168 ppm REC = 0.57 $\overline{D}_A$ = 1.83 mm		
2	0.1% Ti + 0.1% Zr	[T.O] = 145 ppm [T.N] = 65 ppm REC = 0.55 $\overline{D}_A$ = 0.66 mm T. N_V = 0.72*10^6 mm^{-3} $\bar{d}$ = 1.06 μm		
3	0.1% Ti + 0.1% Zr	[T.O] = 148 ppm [T.N] = 248 ppm REC = 0.57 $\overline{D}_A$ = 0.30 mm T. N_V = 1.54*10^6 mm^{-3} $\bar{d}$ = 0.85 μm		
4	0.1% Ti + 0.1% Zr	[T.O] = 139 ppm [T.N] = 490 ppm REC = 0.66 $\overline{D}_A$ = 0.26 mm T. N_V = 5.25*10^6 mm^{-3} $\bar{d}$ = 0.54 μm		
5	0.1% Ti + 0.1% Ce	[T.O] = 205 ppm [T.N] = 67 ppm REC = 0.44 $\overline{D}_A$ = 2.35 mm T. N_V = 1.32*10^6 mm^{-3} $\bar{d}$ = 1.02 μm		
6	0.1% Ti + 0.1% Ce	[T.O] = 193 ppm [T.N] = 246 ppm REC = 0.49 $\overline{D}_A$ = 2.62 mm T. N_V = 1.44*10^6 mm^{-3} $\bar{d}$ = 1.00 μm		

Reference sample no addition

Ti-Zr addition increased N content

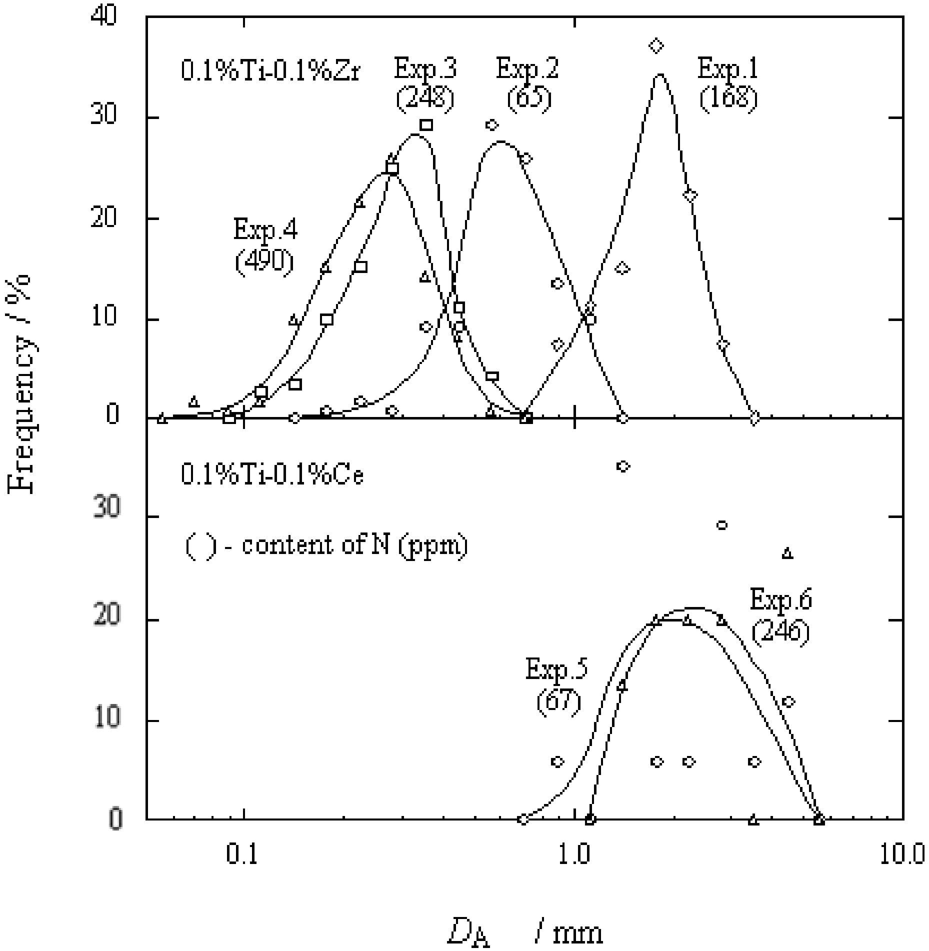
Ti-Ce addition increased N content



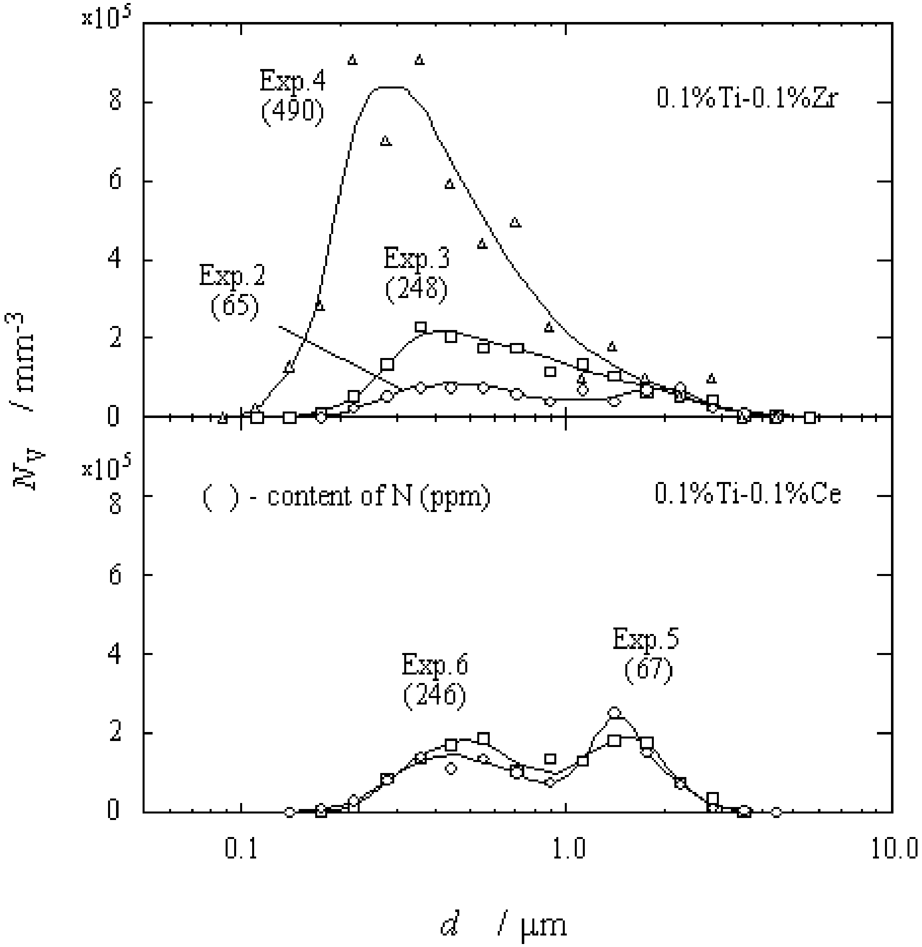


Particles effect on grain size

Grain size distribution



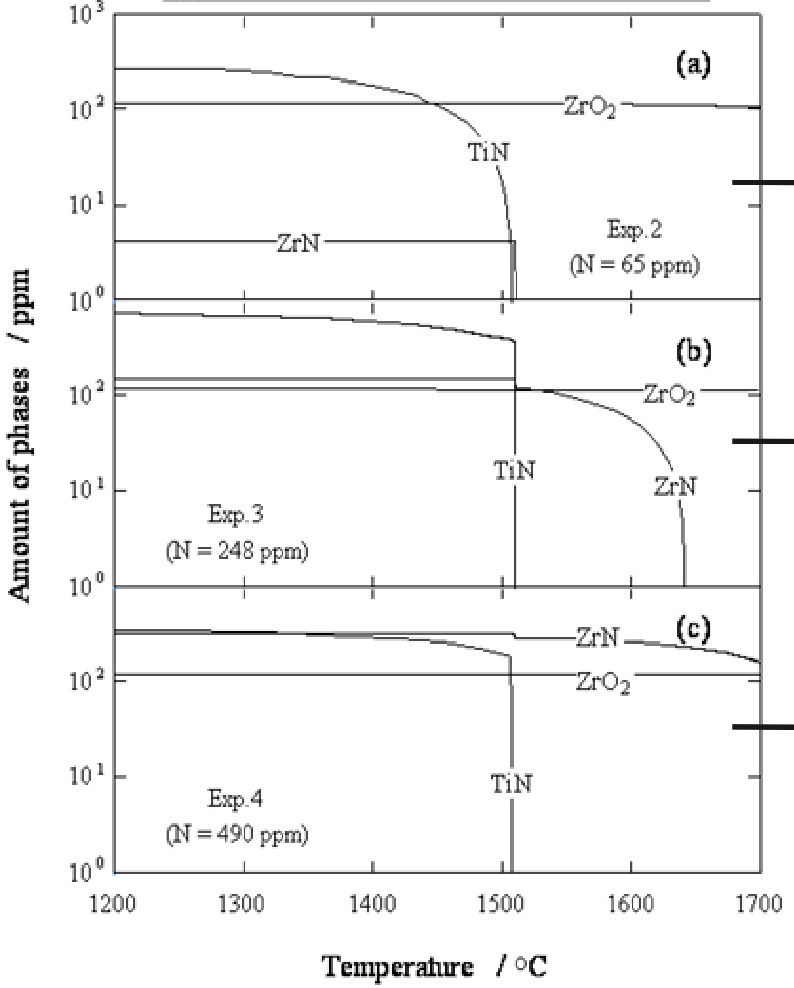
Particles size distribution

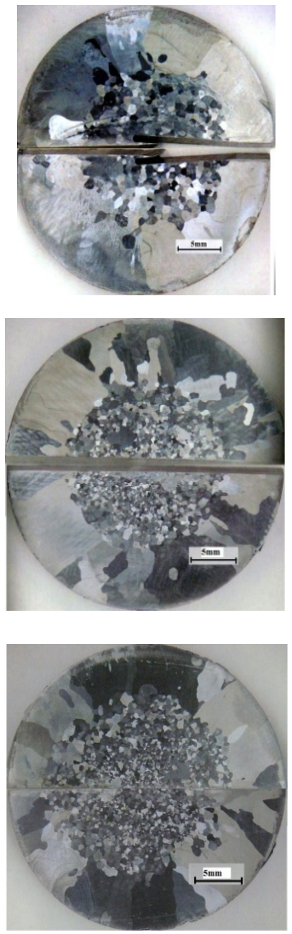


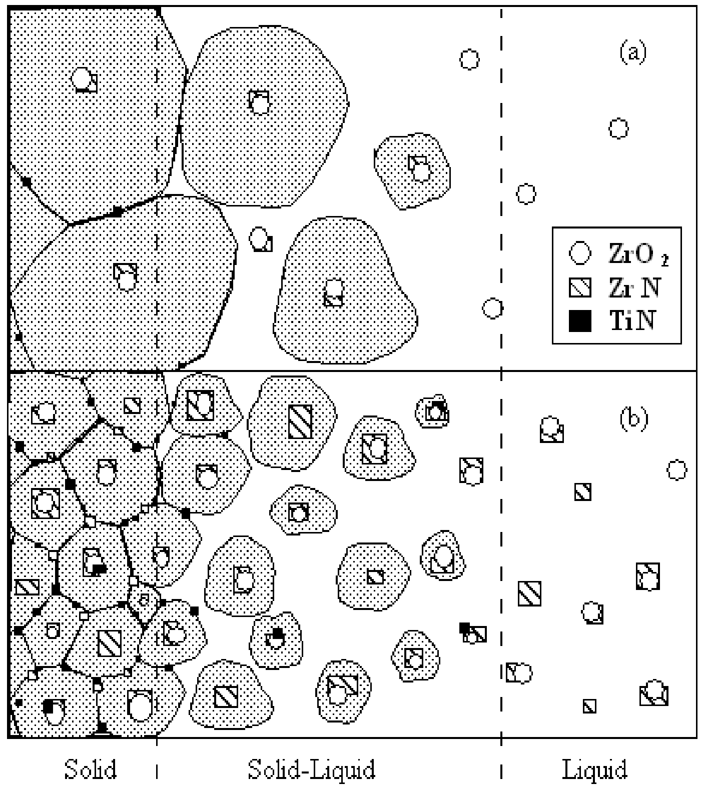
Precipitation, nucleation and pinning

Input data to Thermo-Calc program:

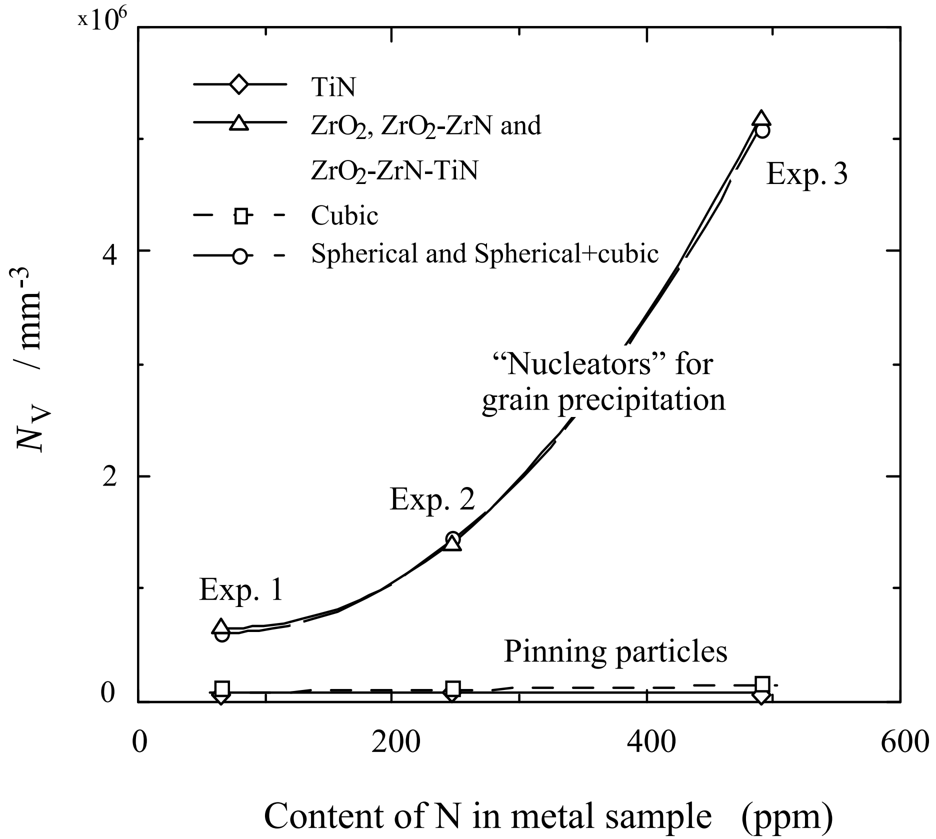
Experiment		O	N	Ti	Zr	Cr	Fe	Pressure
		(mass ppm)				(mass%)		(Pa)
(a)	Exp.2	30	65	652	89	20	balance	1×10⁵
(b)	Exp.3	30	248	588	214	20	balance	1×10⁵
(c)	Exp.4	30	490	249	345	20	balance	1×10⁵

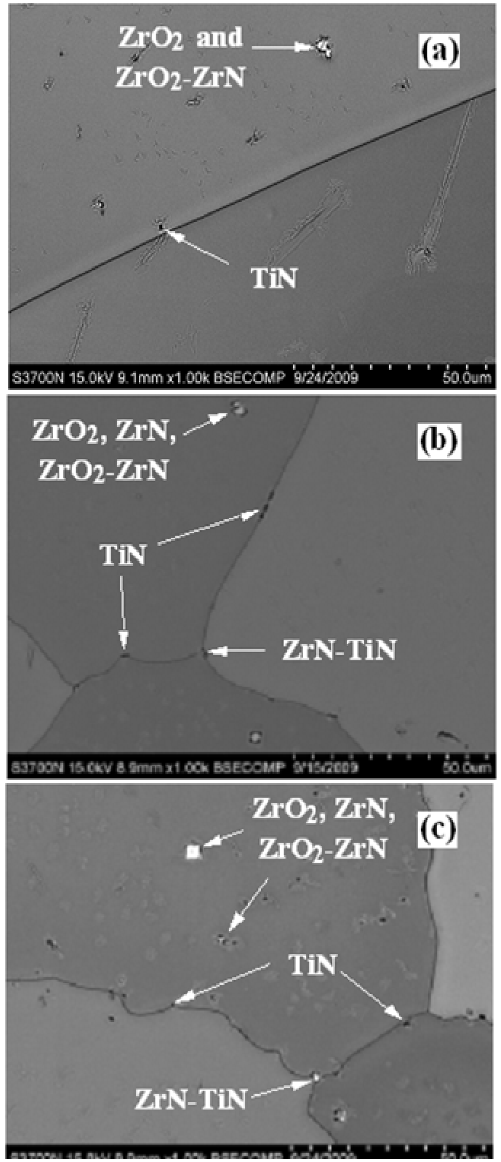




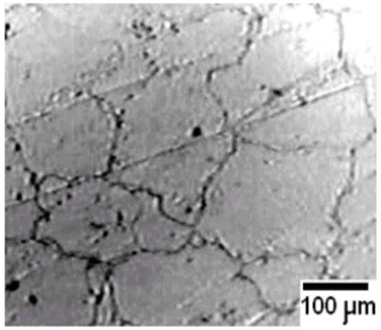
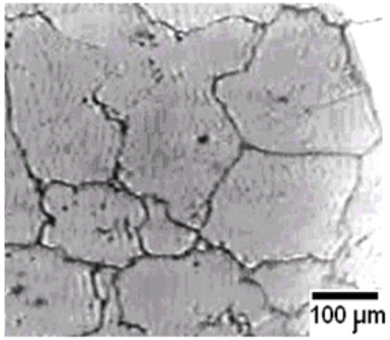
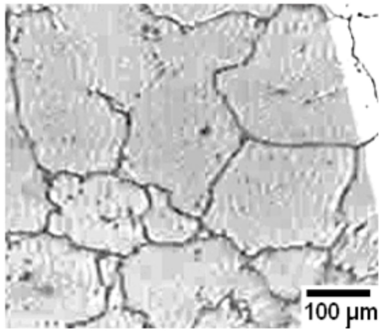
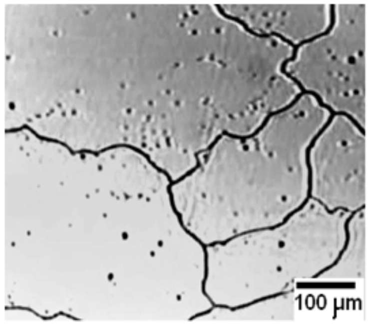
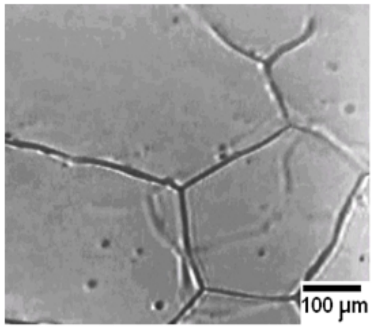
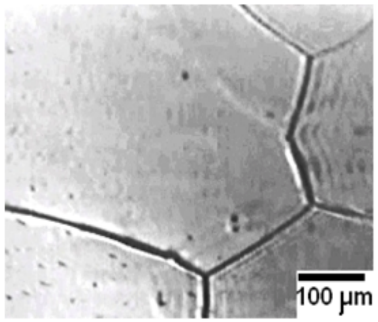


Precipitation and nucleation

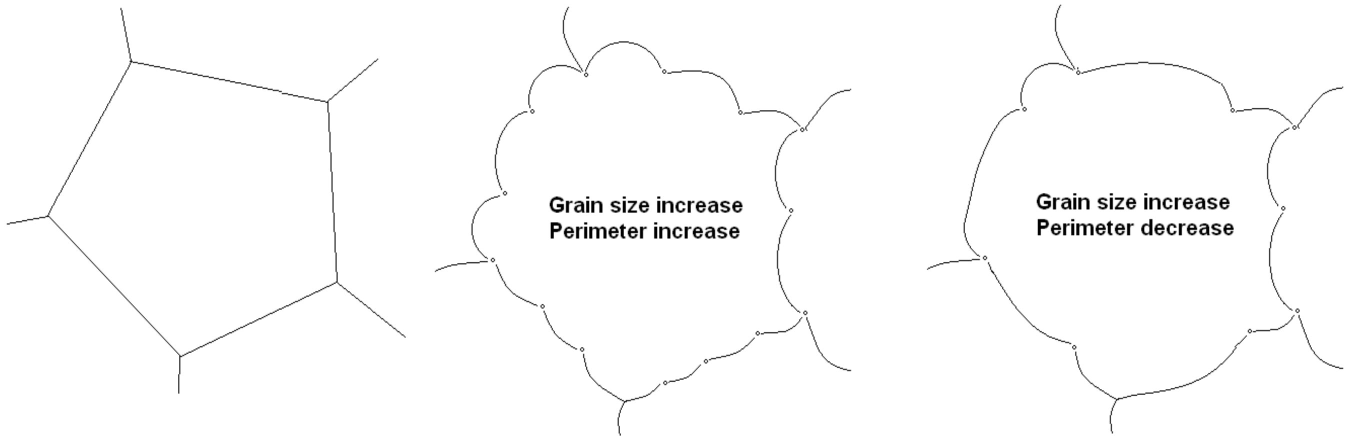


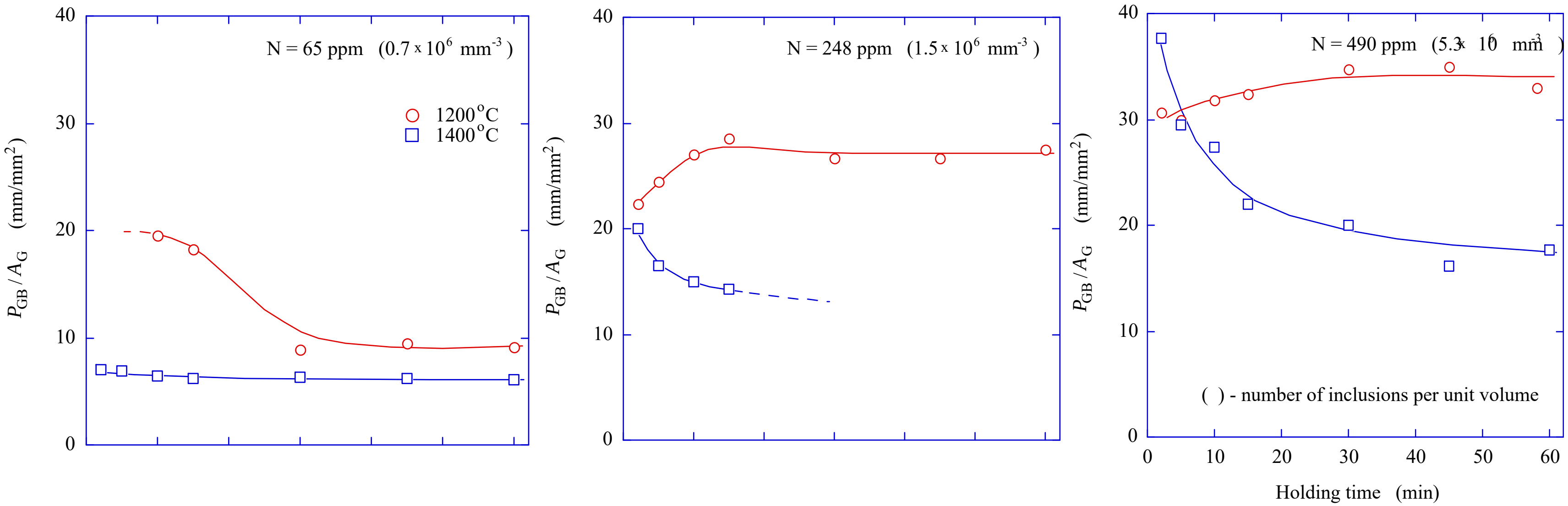


Pinning particles

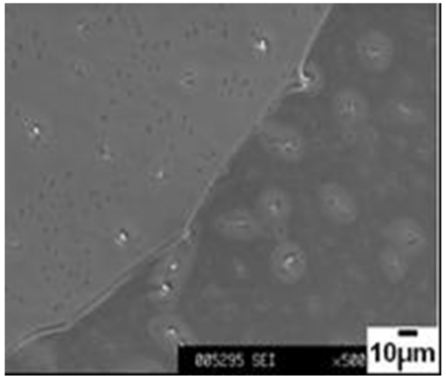
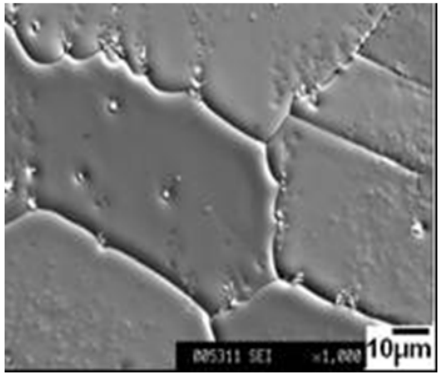
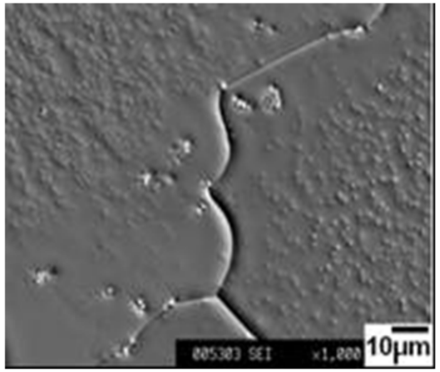
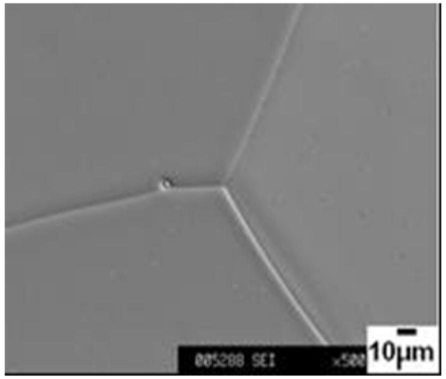
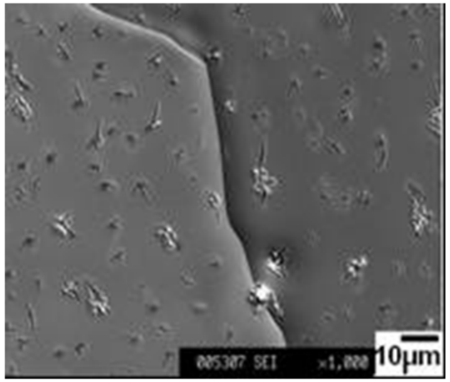
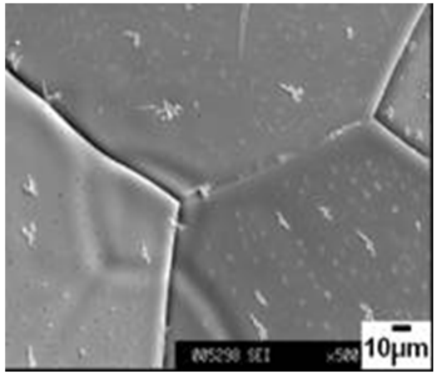
T (°C)	Holding time (min)		
	2	15	60
1200			
1400			

Pinning particles

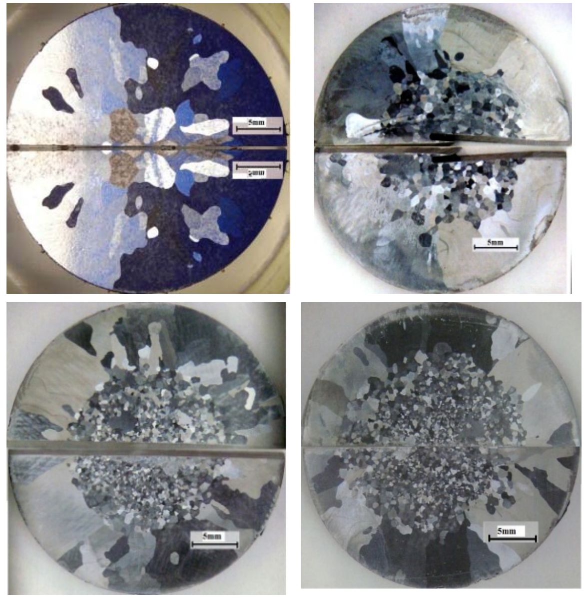




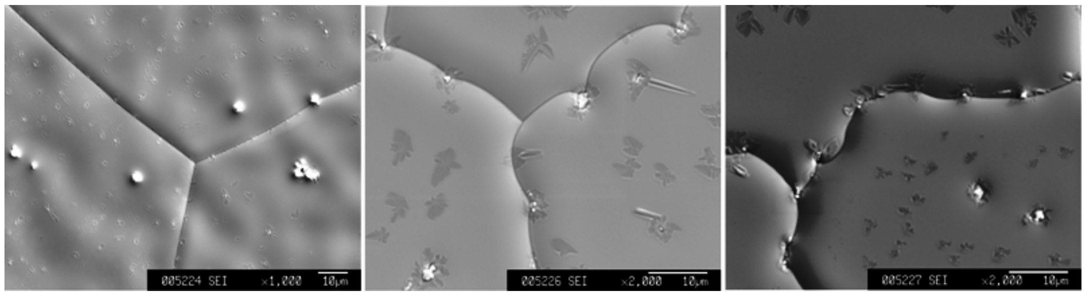
Pinning particles

T (°C)	Nitrogen content (ppm) 65	248	490
1200			
1400			

Particles effect

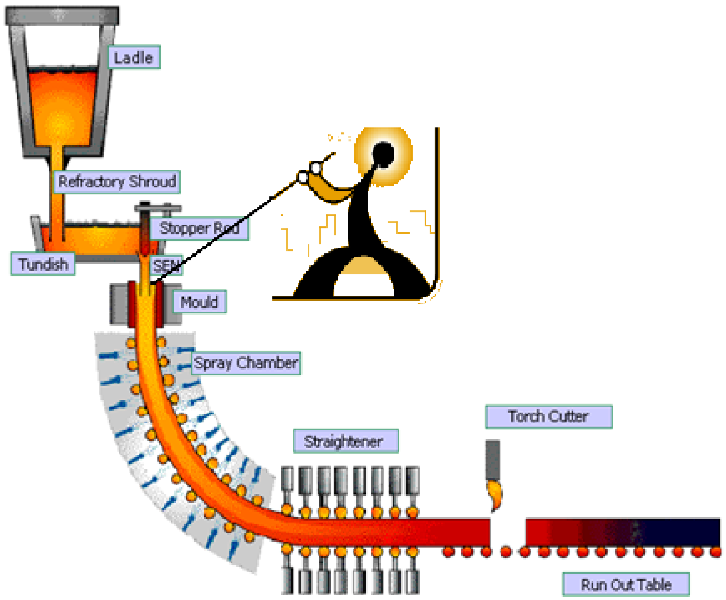


- Solidification stage
  - ✓ Nucleation of grains
- Cooling stage
  - ✓ Secondary precipitation near grain boundaries
  - ✓ Growth speed reduction during cooling
- Welding stage/heat treatment
  - ✓ Precipitation near grain boundaries
  - ✓ Pinning of grain growth



Industrial aspects

- Create a thermodynamic window with respect to O and N for reaction with additives
- Possibilities to reduce alloying addition
- Improve mechanical properties of existing alloys
- Small addition of expensive strong reactants
- Late addition due to high reaction rates
- Late addition due to growth and agglomeration
- Mould metallurgy
- Reduce clogging
- Addition methods



Thank you for your attention!

Tack för att ni lyssnade!

Kiitos mielenkiinnostanne!

Takk for at dere har lyttet!

Danke für Ihre Aufmerksamkeit!

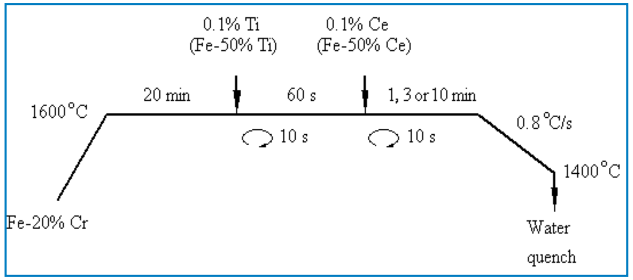
Reservslide

- Nästa slide är en reservslide

Electrolytic extraction of Fe-20mass% Cr

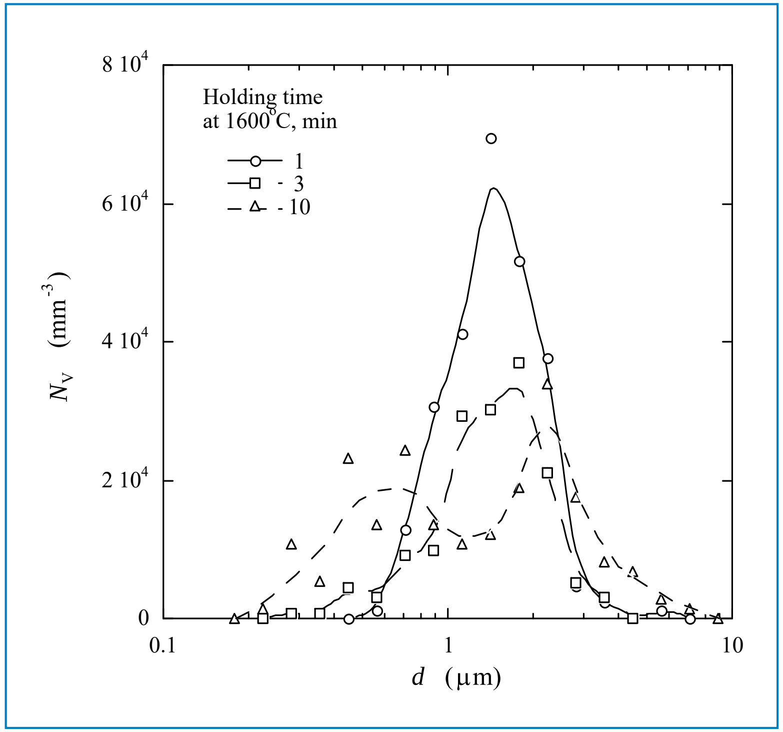
Process

- Deoxidation with Ti and Ce for particles precipitation
- 1, 3 and 10 min holding time
- Tuning of electrolytic parameters
- Avoiding precipitation of Cr-rich layer and dissolution of particles

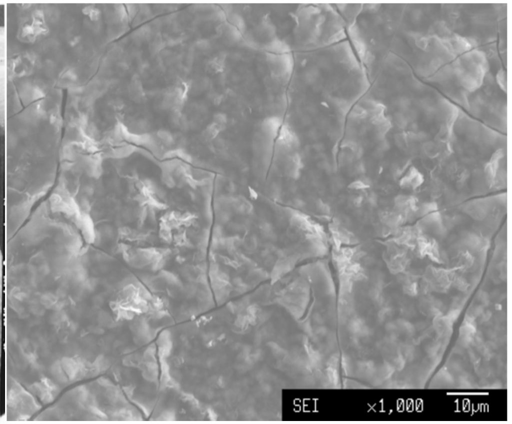


Results

- Strong electrolyte 10%AA, and short time for extraction
- 1 min of holding time is sufficient for precipitation and avoiding of clustering



surface dissolution of Ce-base particles. covering filter



precipitated Cr-rich compound