Microstructure and Mechanical Properties of Directionally Solidified Ti-Fe Eutectic Alloy

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Campinas, SP, Brazil

University of Campinas

- Founded in 1966
- Strong tradition in education and in scientific research (15% of the Brazilian Scientific Production)
- 17,000 undergraduate and 16,000 graduate students

Outline

- Eutectic Alloys
- Eutectic Growth in Metallic Systems
- Eutectic Arrangements
  - Previous Studies:
    - Ni-Si; Al-Nb; Ni-Al-Mo; Al-Nb-Ni
- Ti-Fe Eutectic Alloy
- Results – D.S. of Ti-Fe Eutectic Alloy
  - DS based on Arc Melting Equipment
  - Microstructure
  - Mechanical Behavior
  - Conclusions

Eutectic Alloys

- Eutectic alloys allow the developing of in situ composite for structural applications
- This material consists of phases embedded in a matrix that do not dissolve in each other and are physically separated by a sharp interface between them
- This composite material provides the opportunity of merging the properties of distinct constituents into one material.

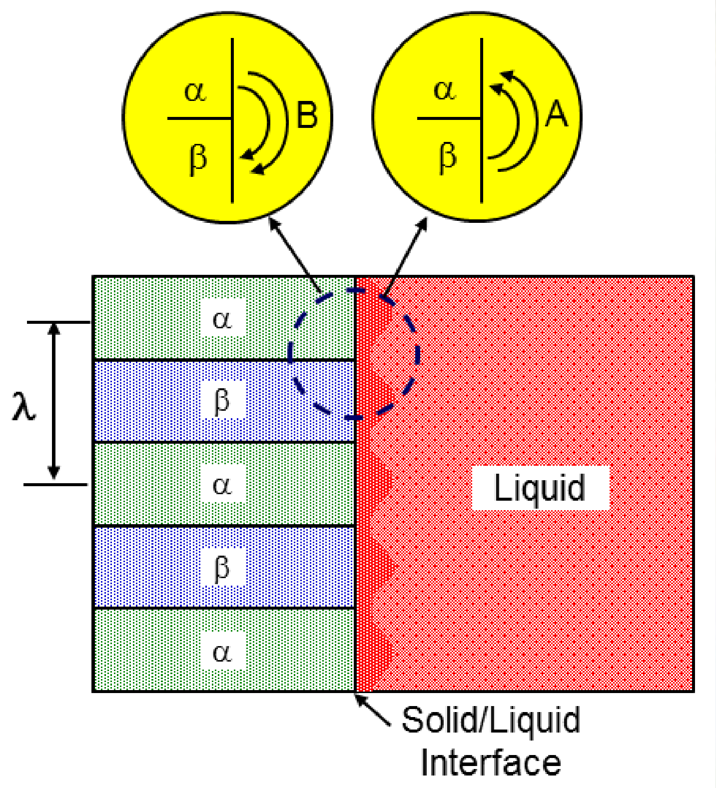
Eutectic Growth

- **Growth of eutectic alloys is an effective method in obtaining in situ composite materials**
- **In situ composites generally have a high degree of thermal stability and improved mechanical properties**
- **D.S. eutectic alloys results in regular structure of two or more solid phases**
- **Eutectic solidification leads to <u>cooperative growth</u>**

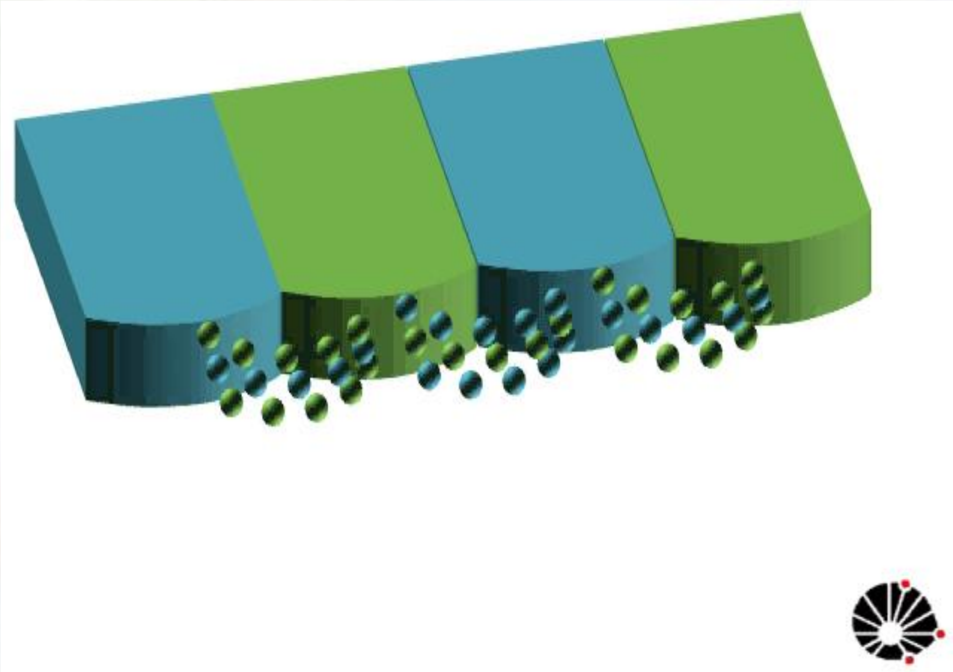
Cooperative Growth

While the α phase segregates B, the β phase rejects A

Such a phenomenon leads to a solute build up in the liquid in front of the α e β phases and hence, to lateral solute diffusion of A and B

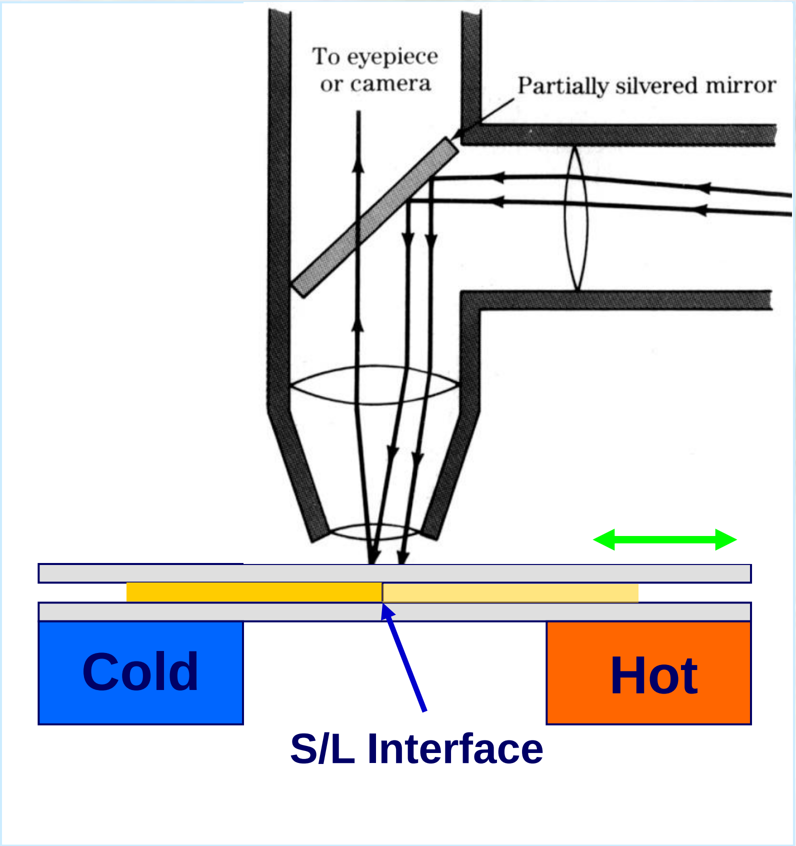


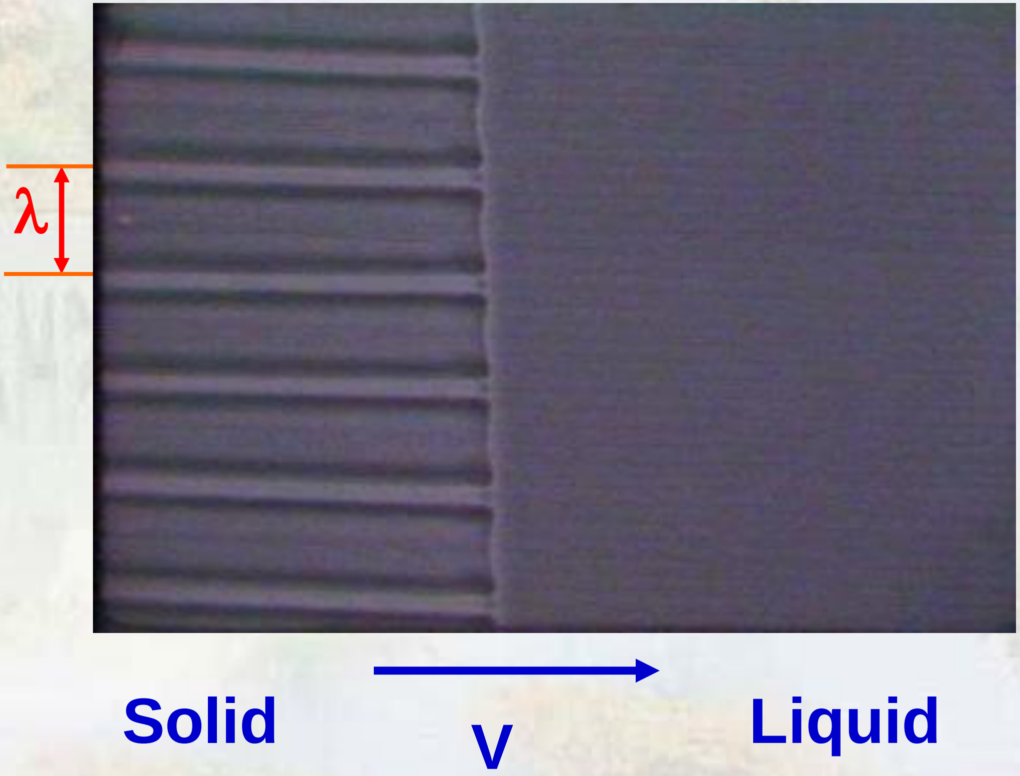
β phase α phase



Eutectic Growth

Growth of CBr_4-C_2Cl_6 eutectic organic alloy





J.D. Hunt and K.A. Jackson – Bell Laboratories – 60's

Previous Studies

Ni-Ni_3Si - Lamellar

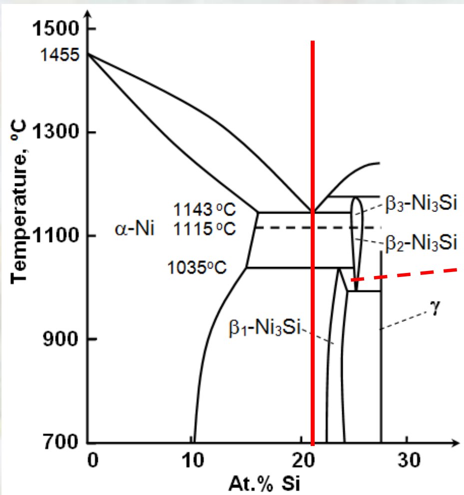
Al_3Nb-Nb_2Al - Lamellar

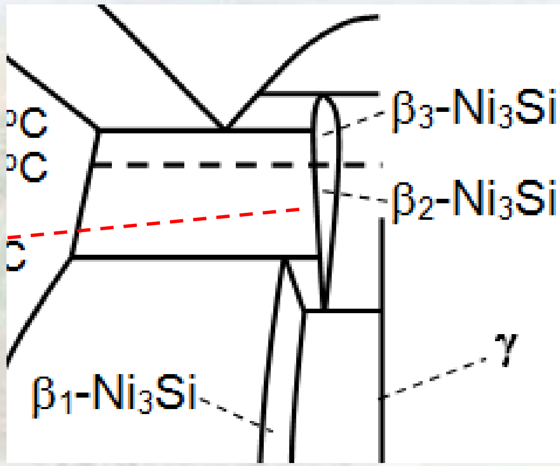
Ni-Al-Mo - Rod-like

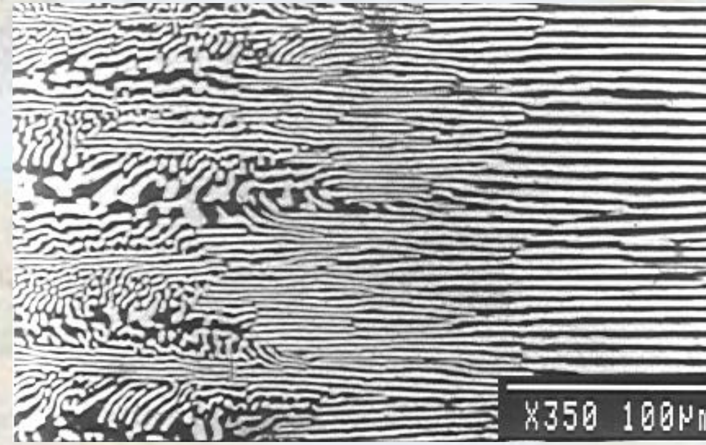
Al_3Nb-Nb_2Al-AlNiNb - Ternary

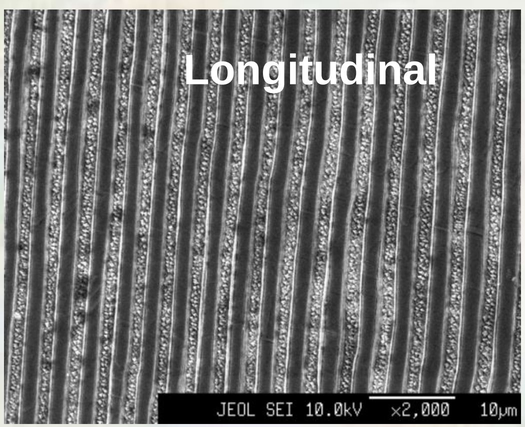
Lamellar Eutectic Growth

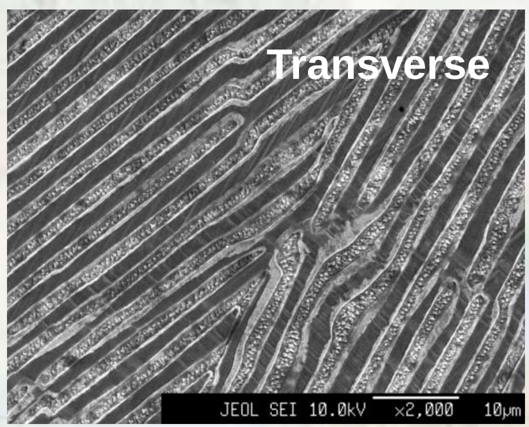
L↔ α(Ni) e $\beta_3(Ni_3Si)$

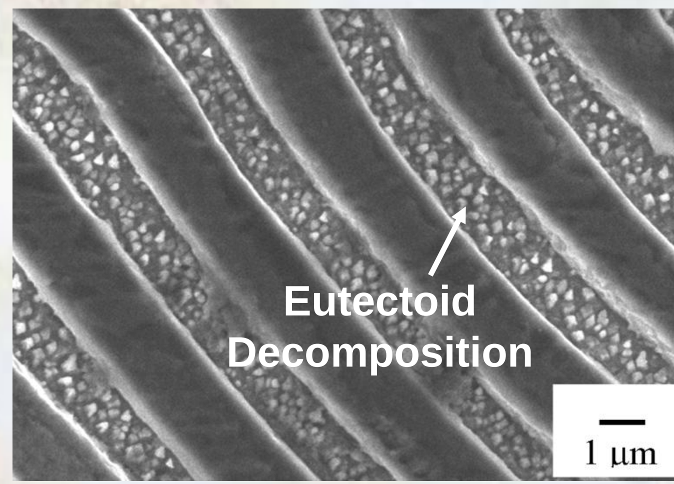










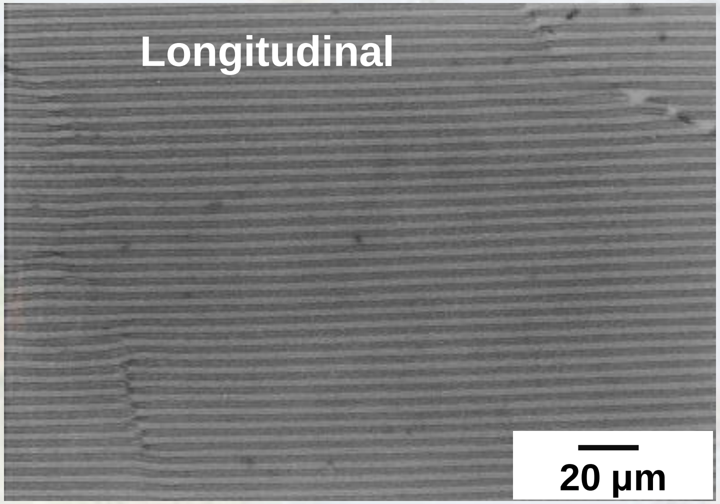


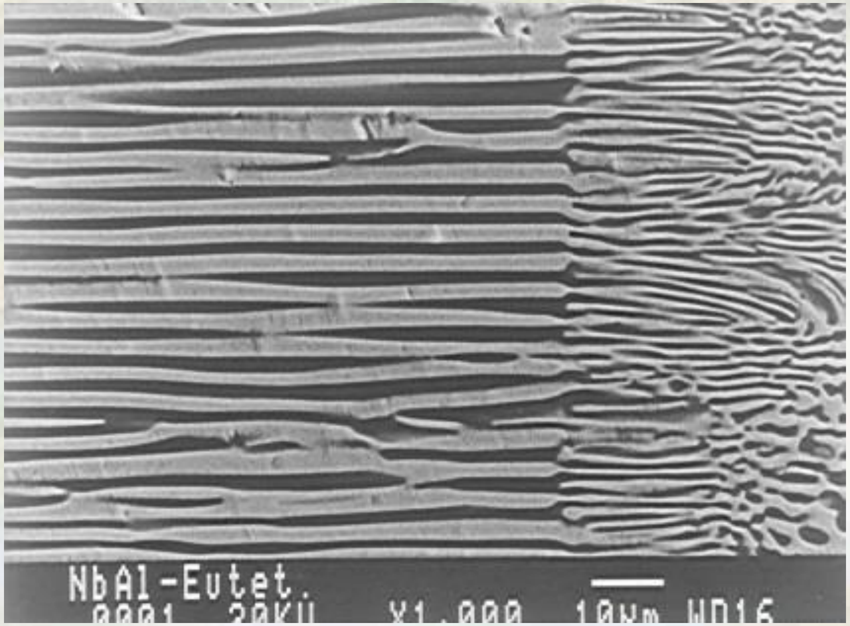
Journal of Crystal Growth 198/199 (1999) 844

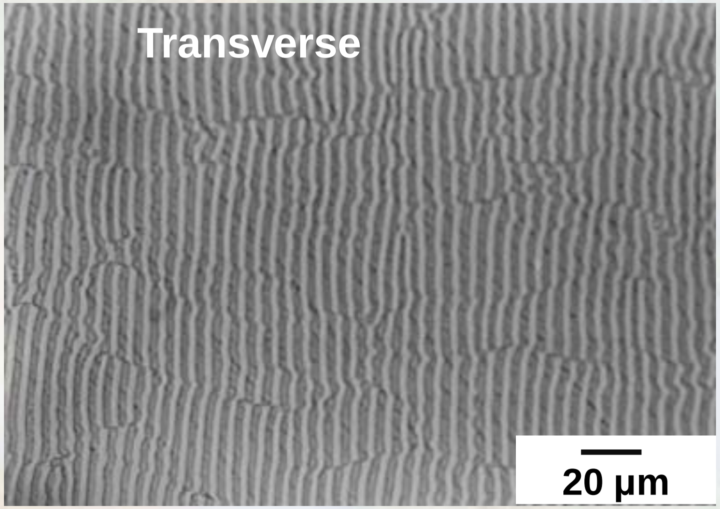
Lamellar Eutectic Growth

L↔ Al_3Nb–Nb_2Al

Eutectic Transformation
1595°C / Al-42.2Nb at%





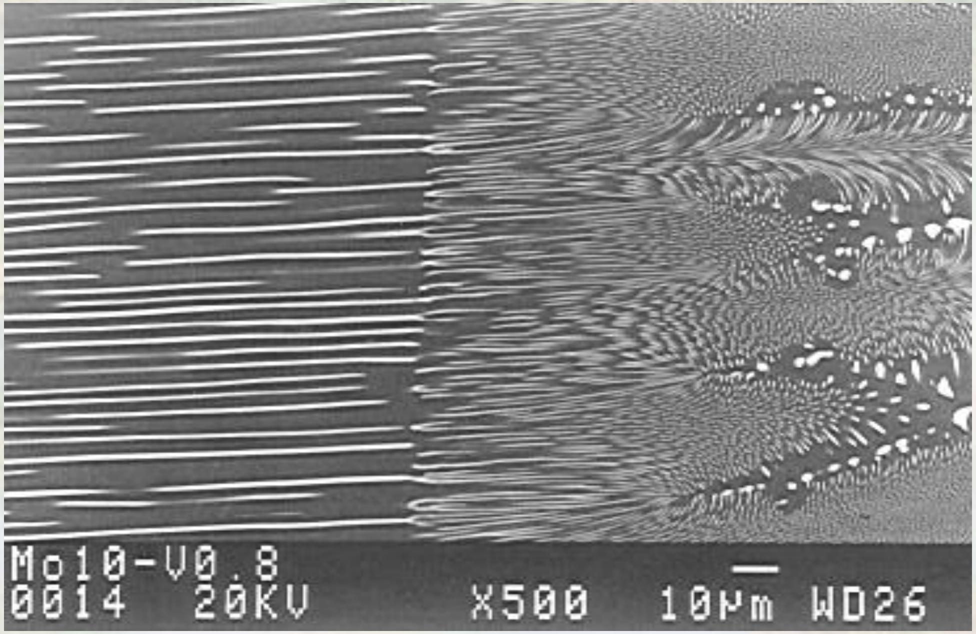


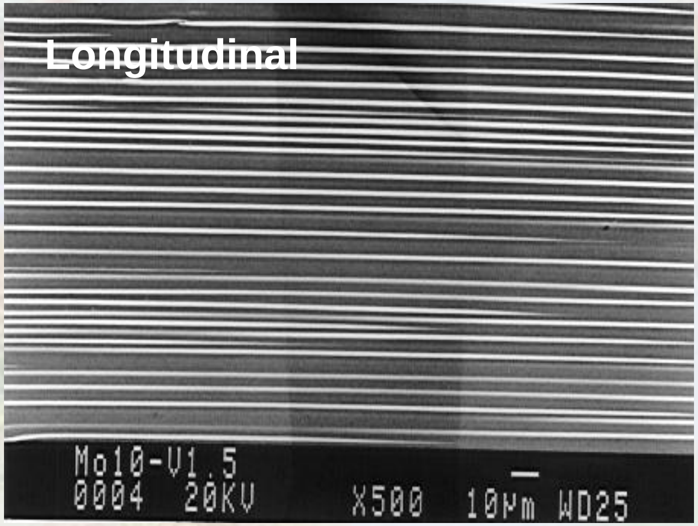
Materials Characterization 54 (2005) 187

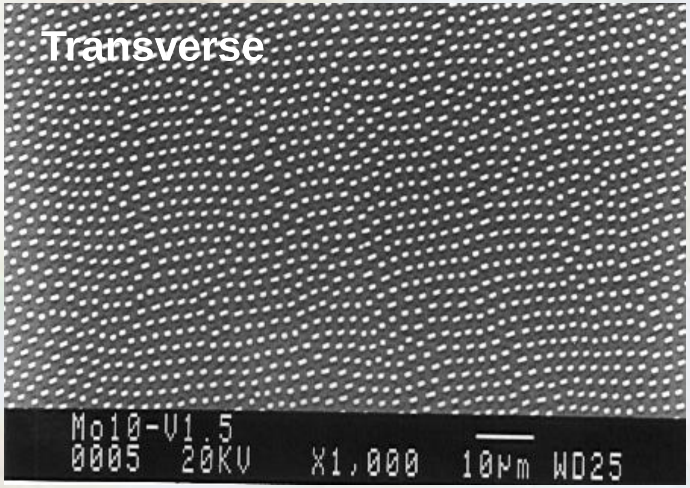
Rod-Like Eutectic Growth

L↔ NiAl–Mo

Eutectic Transformation
1600°C / NiAl-10Mo at%



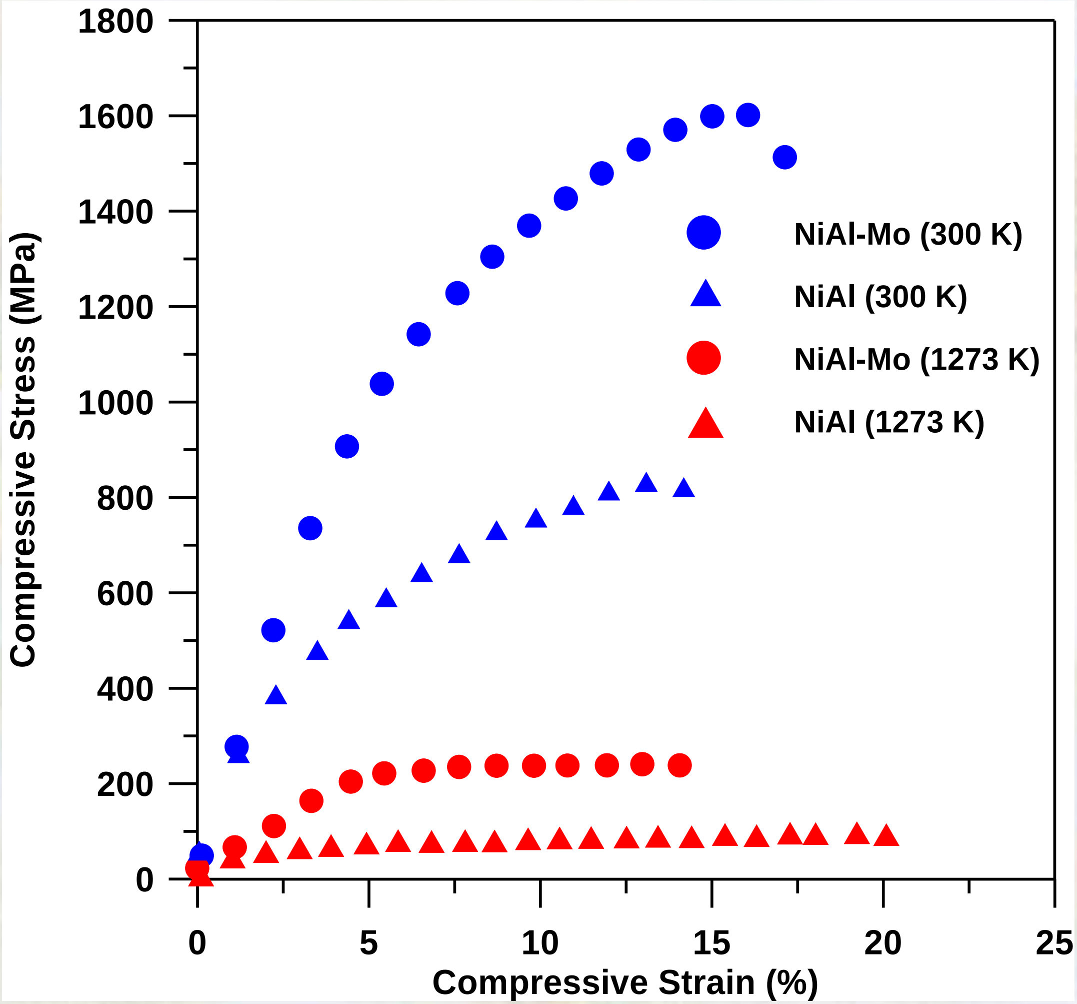




Journal of Alloys and Compounds 381 (2004) 91

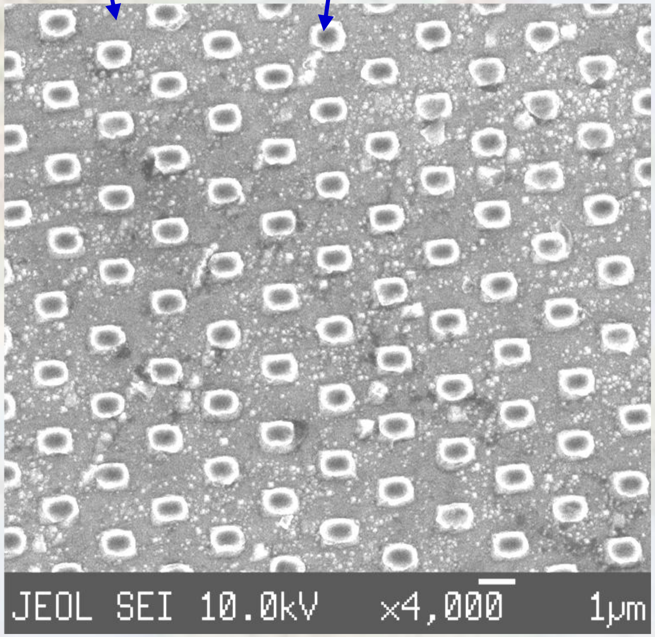
Rod-Like Eutectic Growth

NiAl-Mo In Situ Composite



NiAl

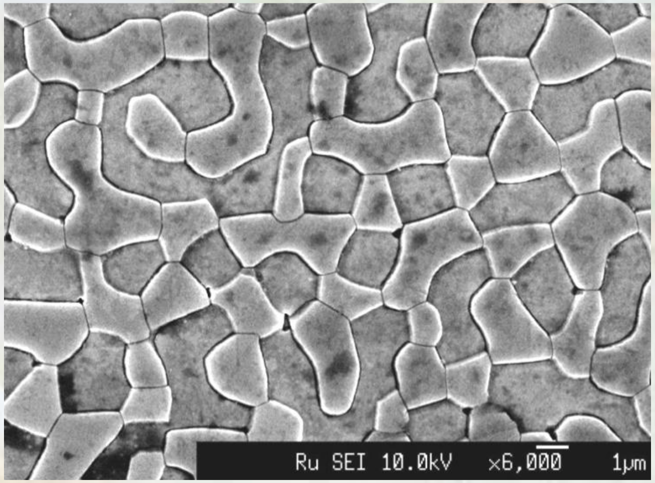
Mo

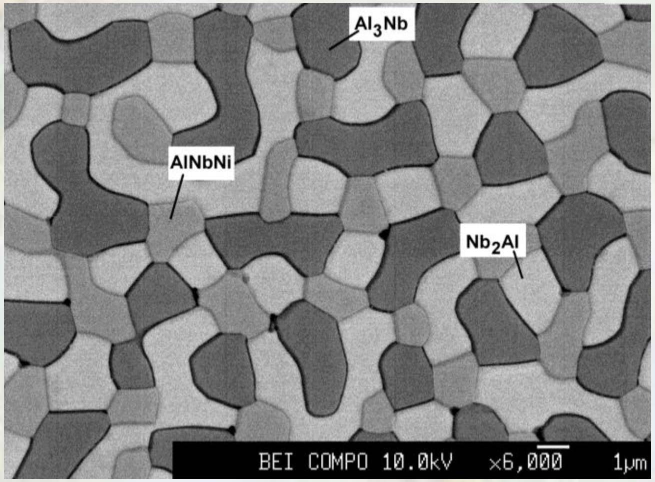


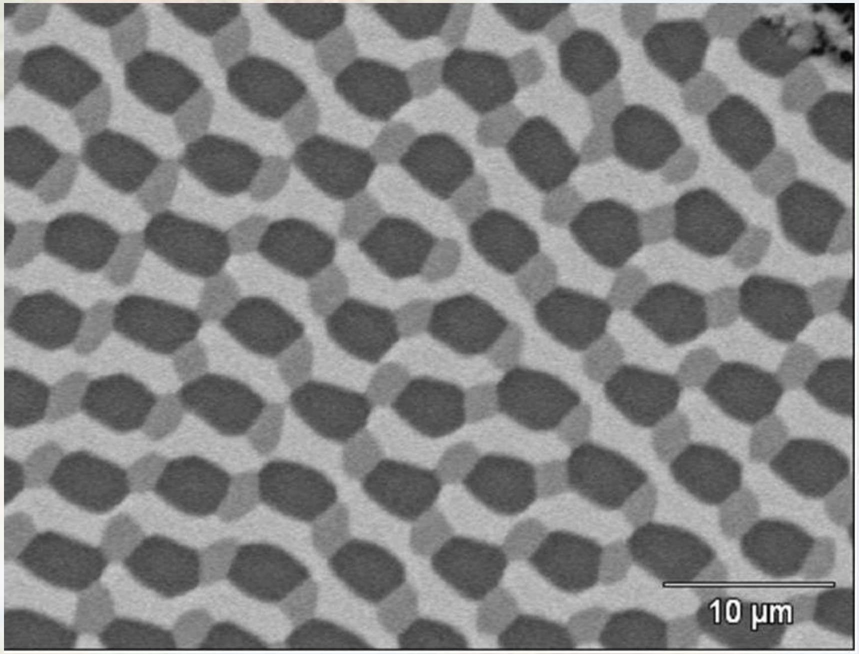
Ternary Eutectic Growth

L↔ $Al_3Nb–Nb_2Al$-AlNbNi

Eutectic Transformation 1520^{o}C / Al-40.4Nb-2.4Ni at%



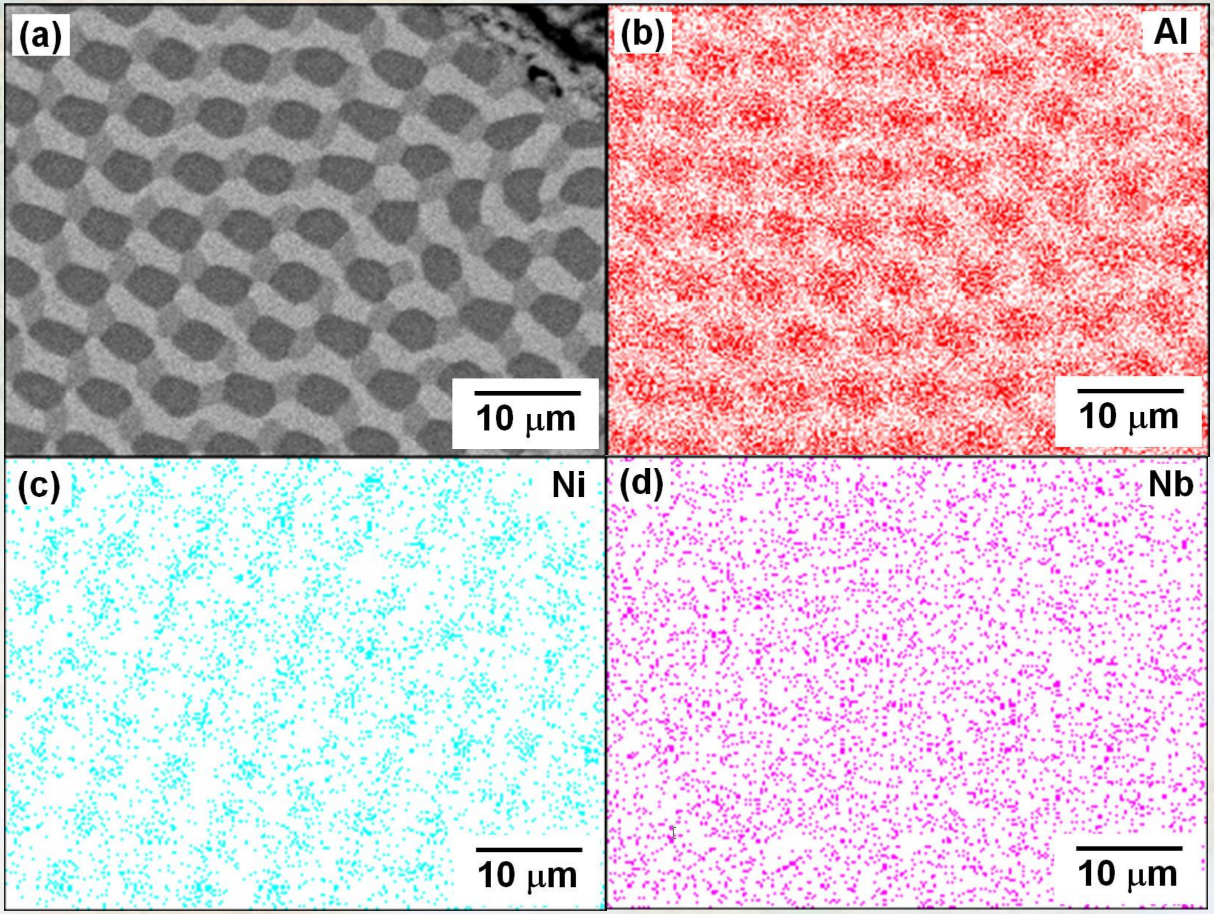




Scripta Materialia 48 (2003) 1495

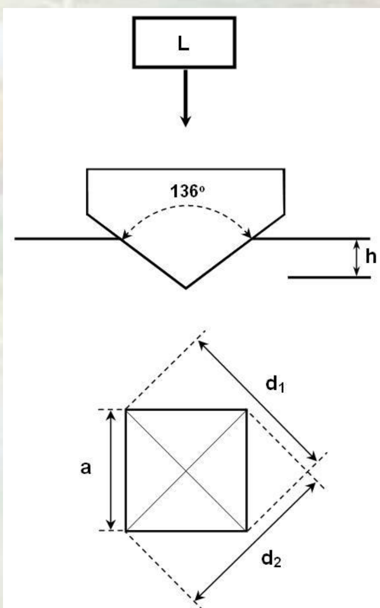
Ternary Eutectic Growth

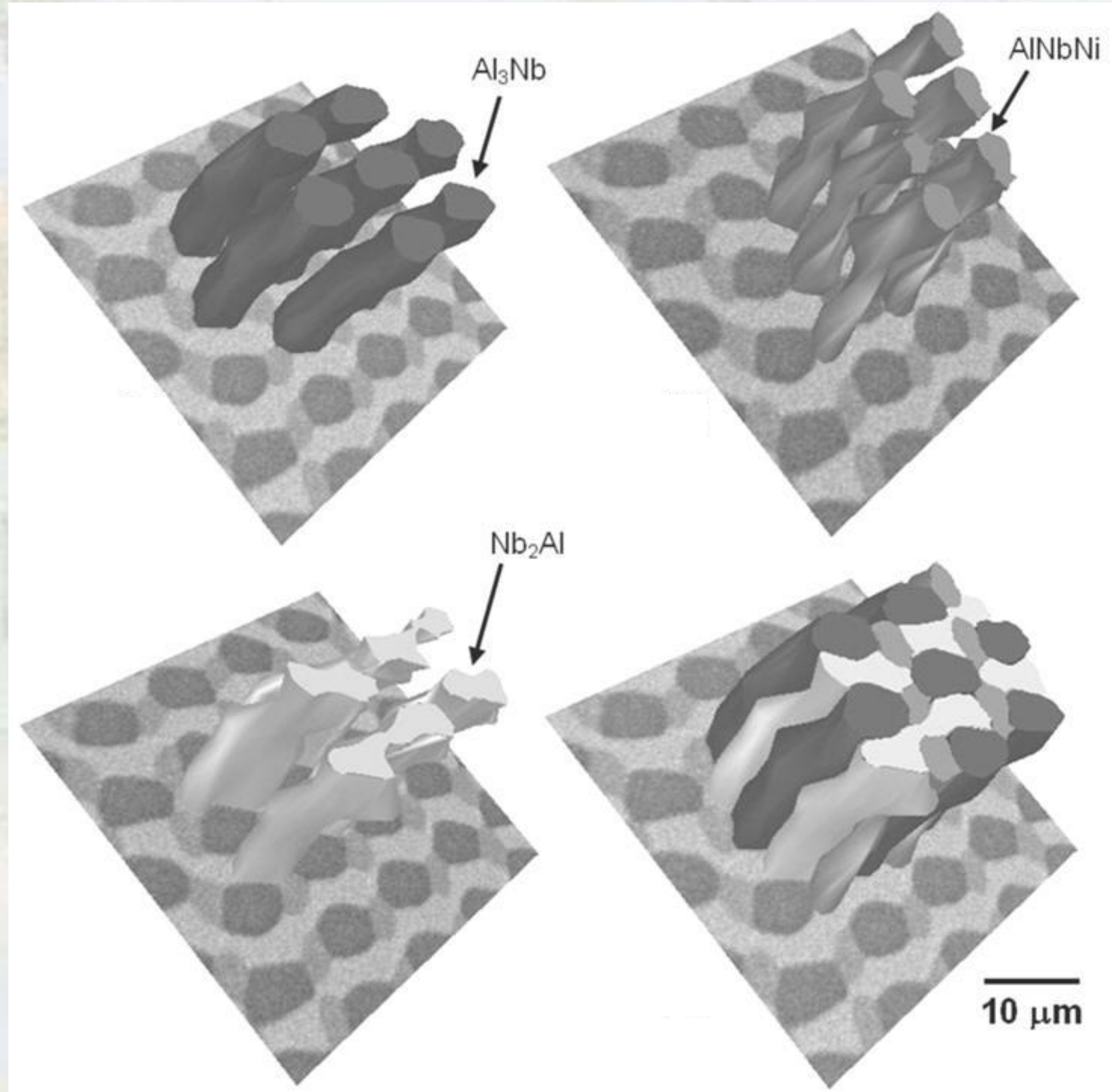
Atom distribution by X-Ray Maps



Ternary Eutectic Growth

3D reconstruction of the ternary eutectic microstructure using the serial sectioning technique





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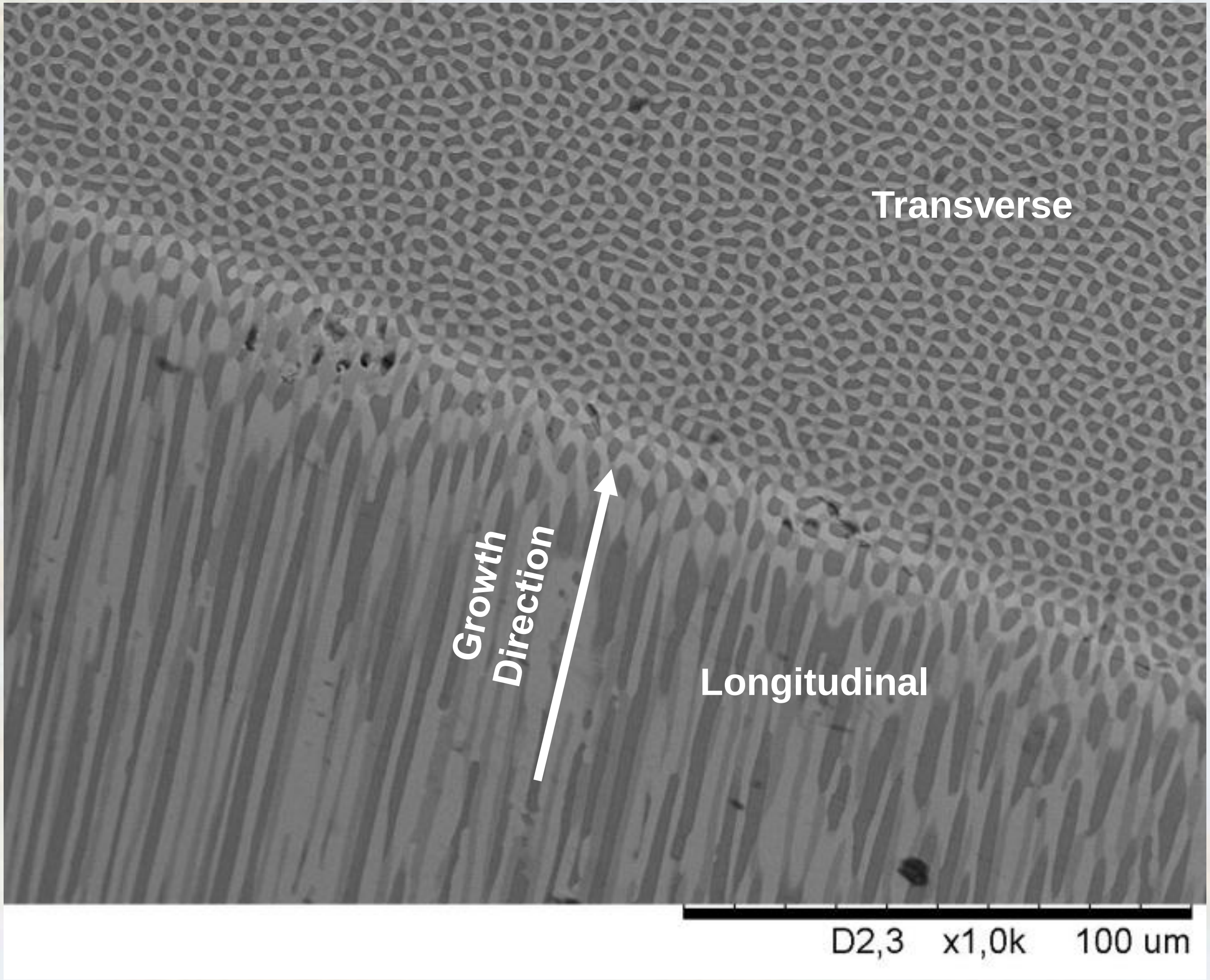
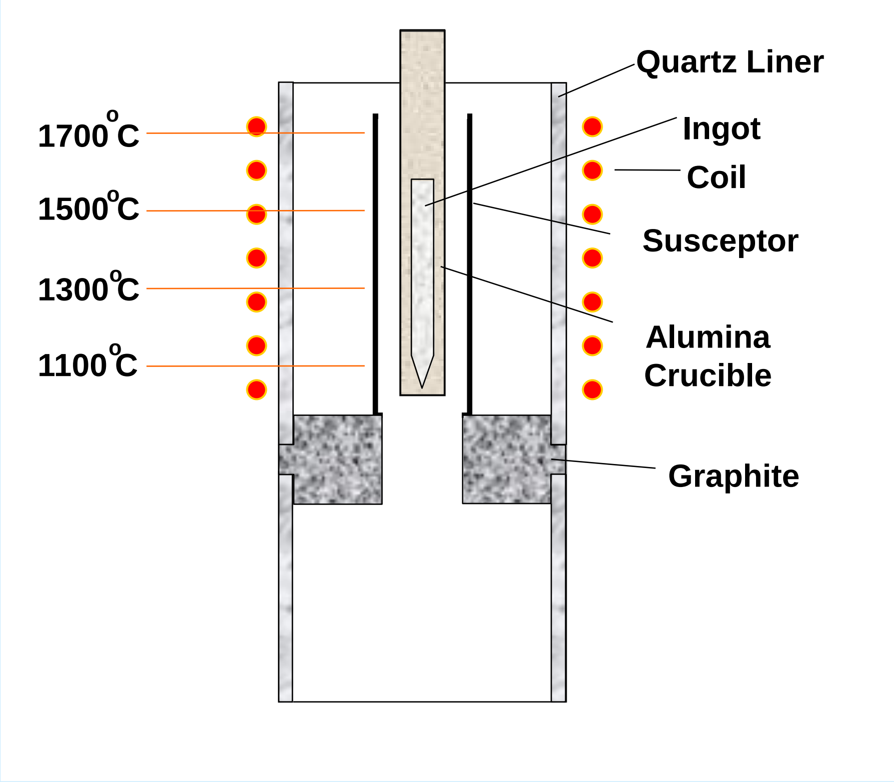
Ternary Eutectic Growth



Materials & Design 33 (2012) 563

Directional Solidification

- D.S. of eutectics was carried out by using a vertical Bridgman furnace, in Al_2O_3 crucibles (0.8 ID x 1.0 cm OD and 6.0 cm long)



- Ti alloys can not be processed in Al_2O_3 crucibles

Ti-Fe System

- **Mechanical performance of Ti can be considerably enhanced by combining it and Fe, causing an eutectic transformation:**

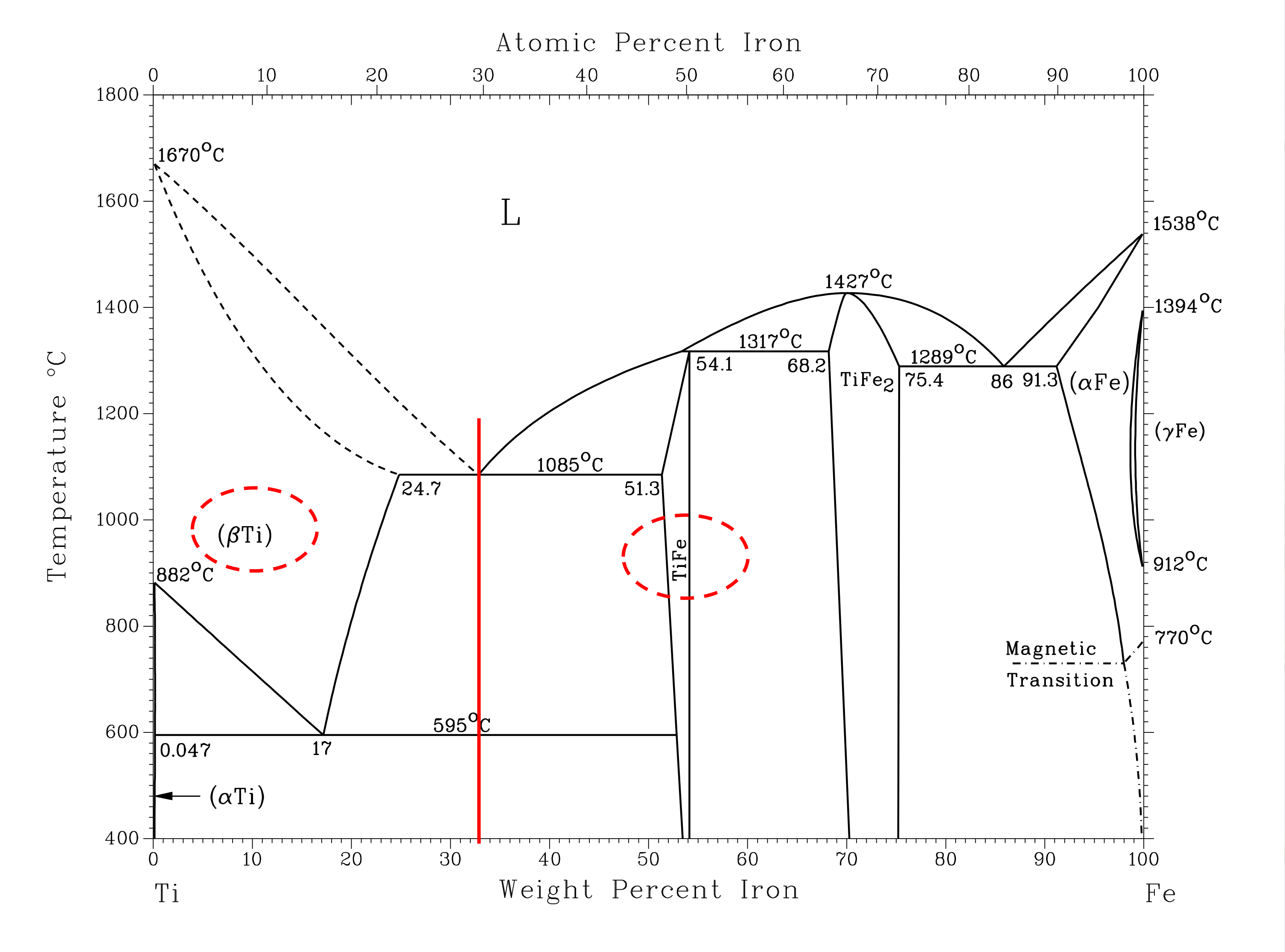
  L↔ βTi–TiFe   1095^{o}C/Ti-32.5Fe wt%

  βTi: ductile BCC phase

  TiFe: high strength phase

- **Directional solidification was carried out in a setup that employs a water-cooled copper crucible combined with a voltaic electric arc moving through the sample.**

Ti-Fe System

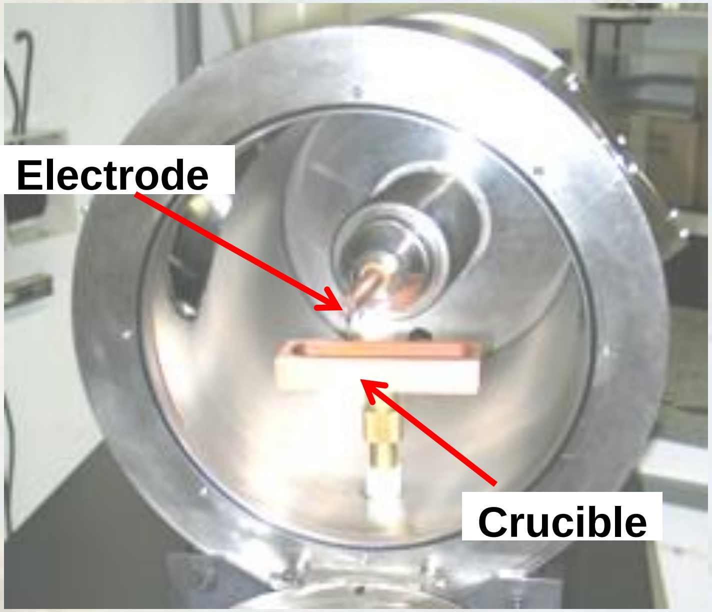


Atomic Percent Iron
0
10
20
30
40
50
60
70
80
90
100
1800
1600
1400
1200
1000
800
600
400
Temperature °C
1670°C
L
1538°C
1427°C
1394°C
1317°C
1289°C
54.1
68.2
TiFe2
75.4
86
91.3
(αFe)
(γFe)
1085°C
24.7
51.3
(βTi)
TiFe
882°C
912°C
770°C
Magnetic
Transition
595°C
0.047
17
(αTi)
Weight Percent Iron
Ti
Fe

Sample Preparation

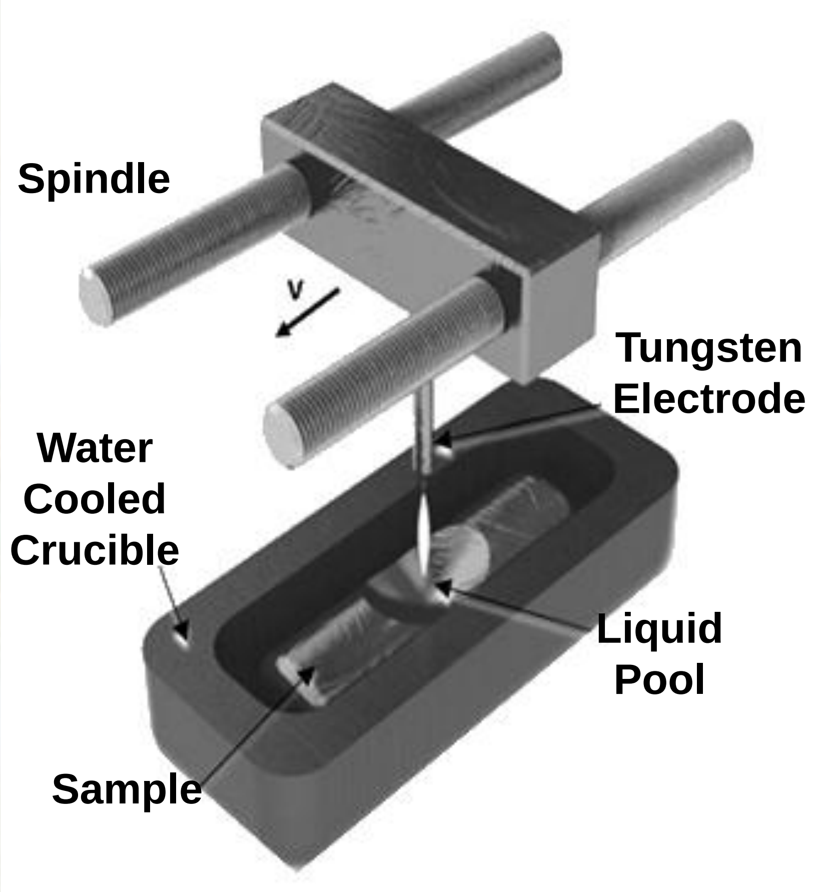
- **Arc furnace with non-consumable W electrode and water cooled copper hearth under Ar atmosphere.**

Nominal (at.%)
Ti-32.5 Fe



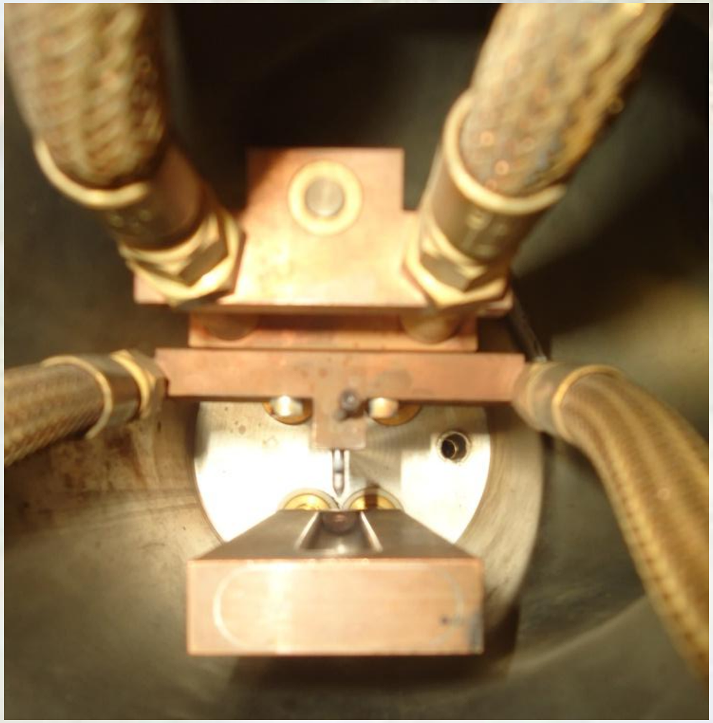
Directional Solidification

- **Arc furnace with a nonconsumable W electrode that moves longitudinally along the ingot at different rates**



Directional Solidification

- **Three solidification rates chosen:**
  V=10, 30 and 60 mm/h.



Sample Characterization

- Chemical composition
  - X-ray fluorescence spectrometry - Rigaku RIX 3100
  - Oxygen and nitrogen - LECO TC-400 analyzer
- Phase transformations
  - Differential thermal analysis - Netzsch STA 409
- Microstructure characterization
  - Scanning electron microscopy - Zeiss EVO 15
  - Transmission electron microscopy - JEOL JEM 2100
  - X-ray diffraction - PANalytical X'Pert
- Mechanical characterization
  - Vickers Hardness test – Buehler 2100
  - Nano-indentation – NHT – CSM Instruments
  - Compressive tests – EMIC DL2000

Chemical Composition

- **Chemical composition:**
  - **X-ray fluorescence spectrometry - Rigaku RIX 3100**
  - **Oxygen and nitrogen - LECO TC-400 analyzer**

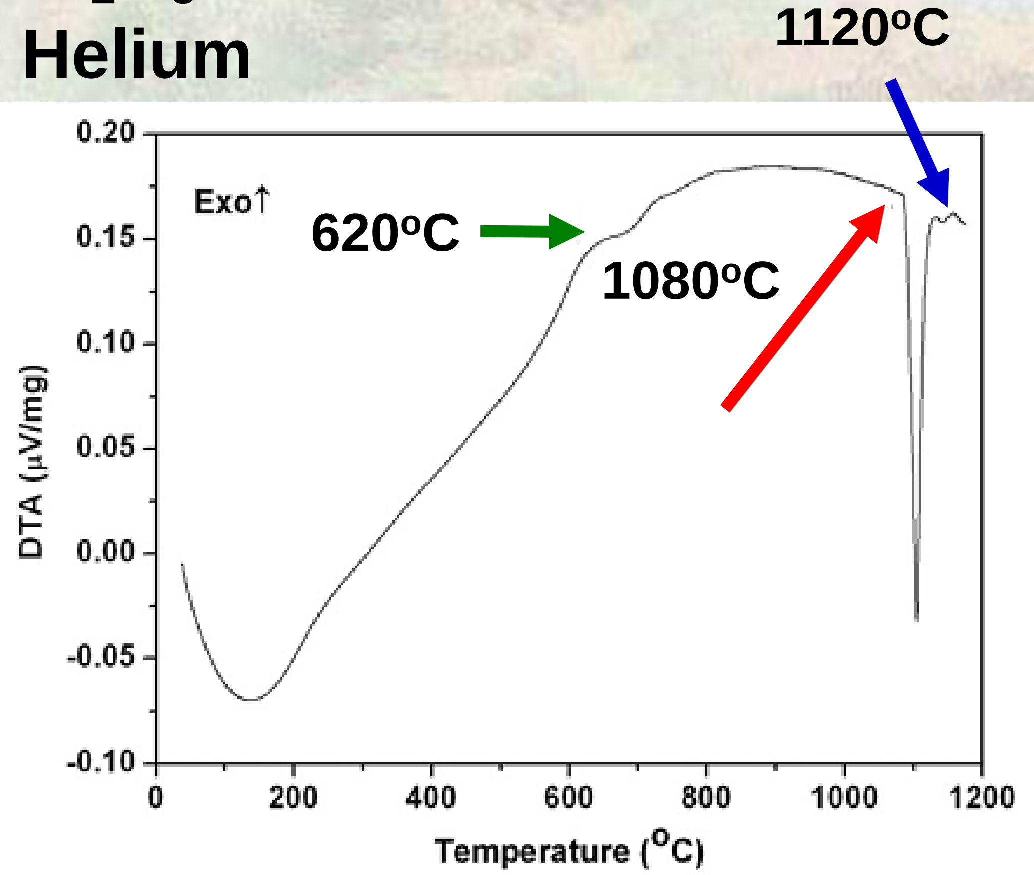
Nominal (at.%)	Measured (at.%)
Ti-32.5 Fe	Ti-32.8 Fe

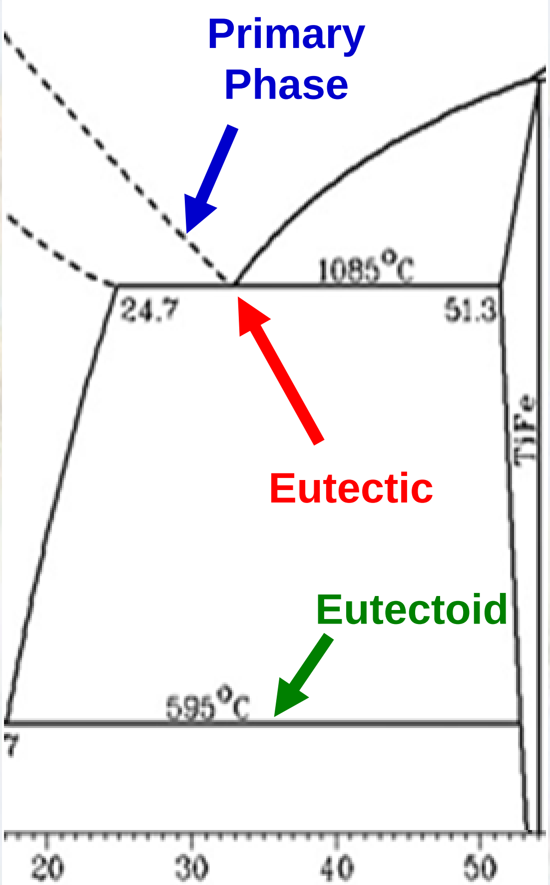
Ti	O (wt.%)	N (wt.%)
Balance	0.0855	0.014725

very low interstitial contamination

Phase Transformations

- DTA - Ti-32.5Fe eutectic alloy
- Heating rate of 10ºC/min
- Al_2O_3 Crucible
- Helium

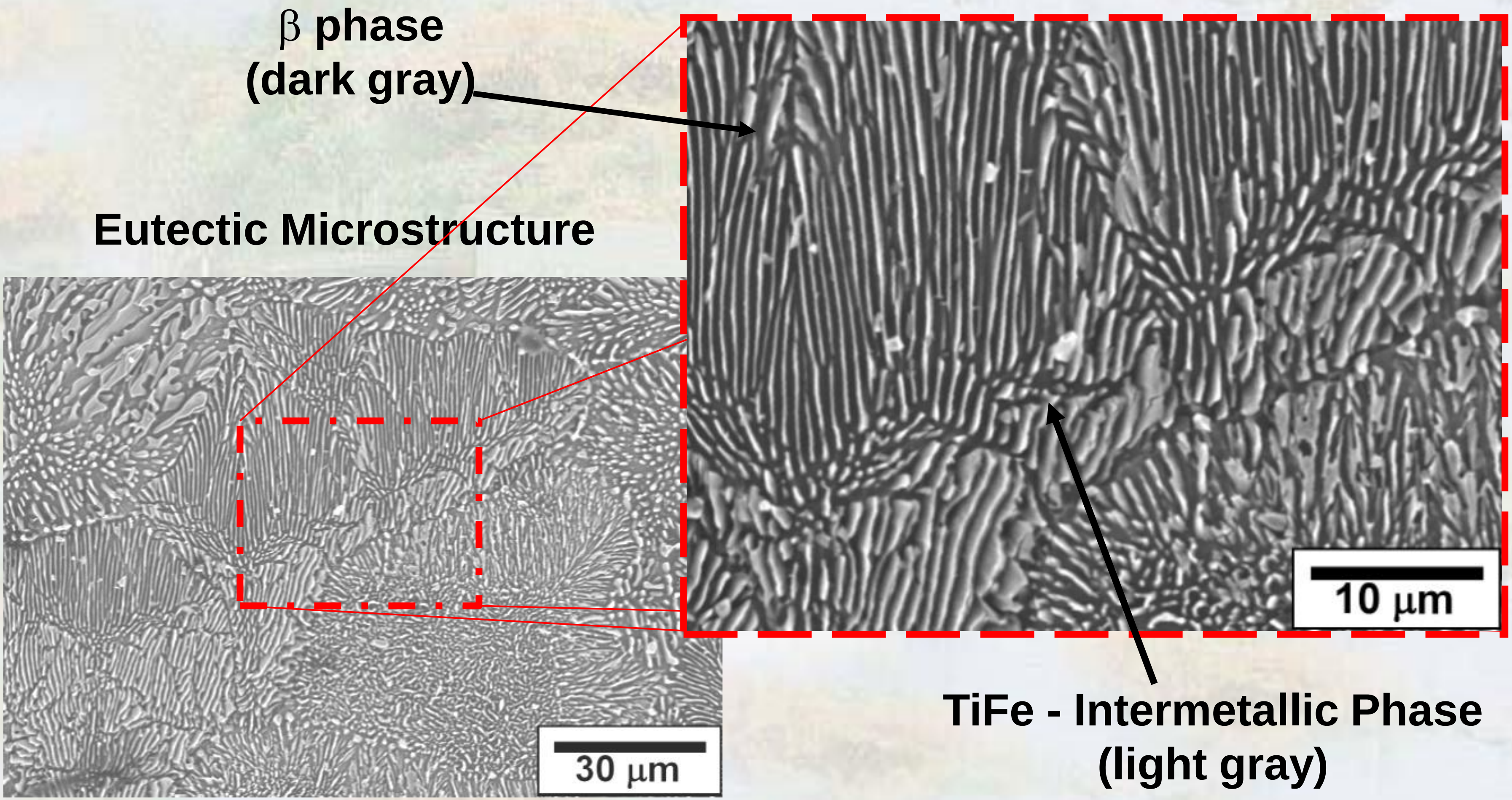




Ti-Fe Phase Diagram

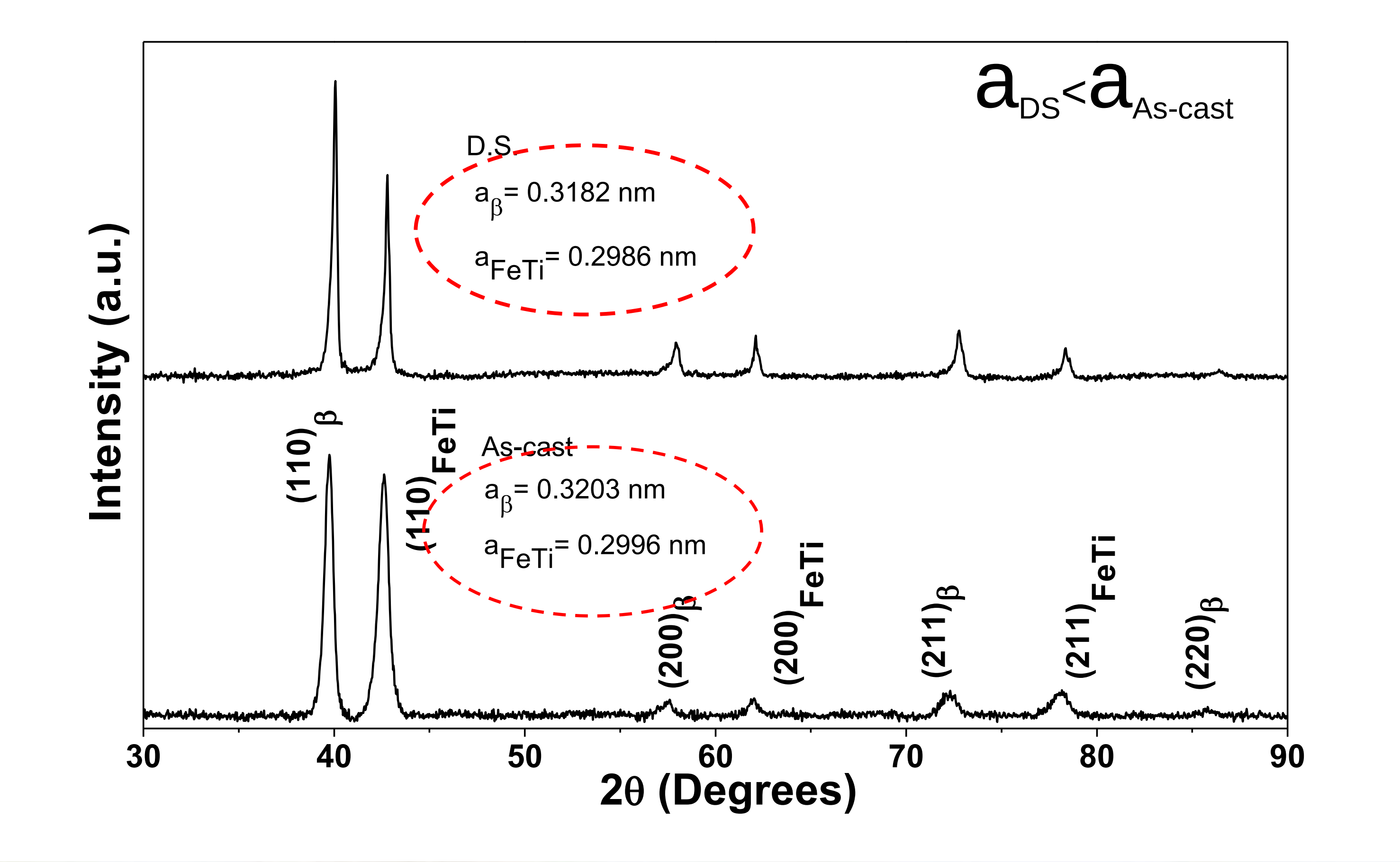
As-Cast Condition

- **SEM micrographs of the Ti–Fe eutectic alloy in the as-cast condition**



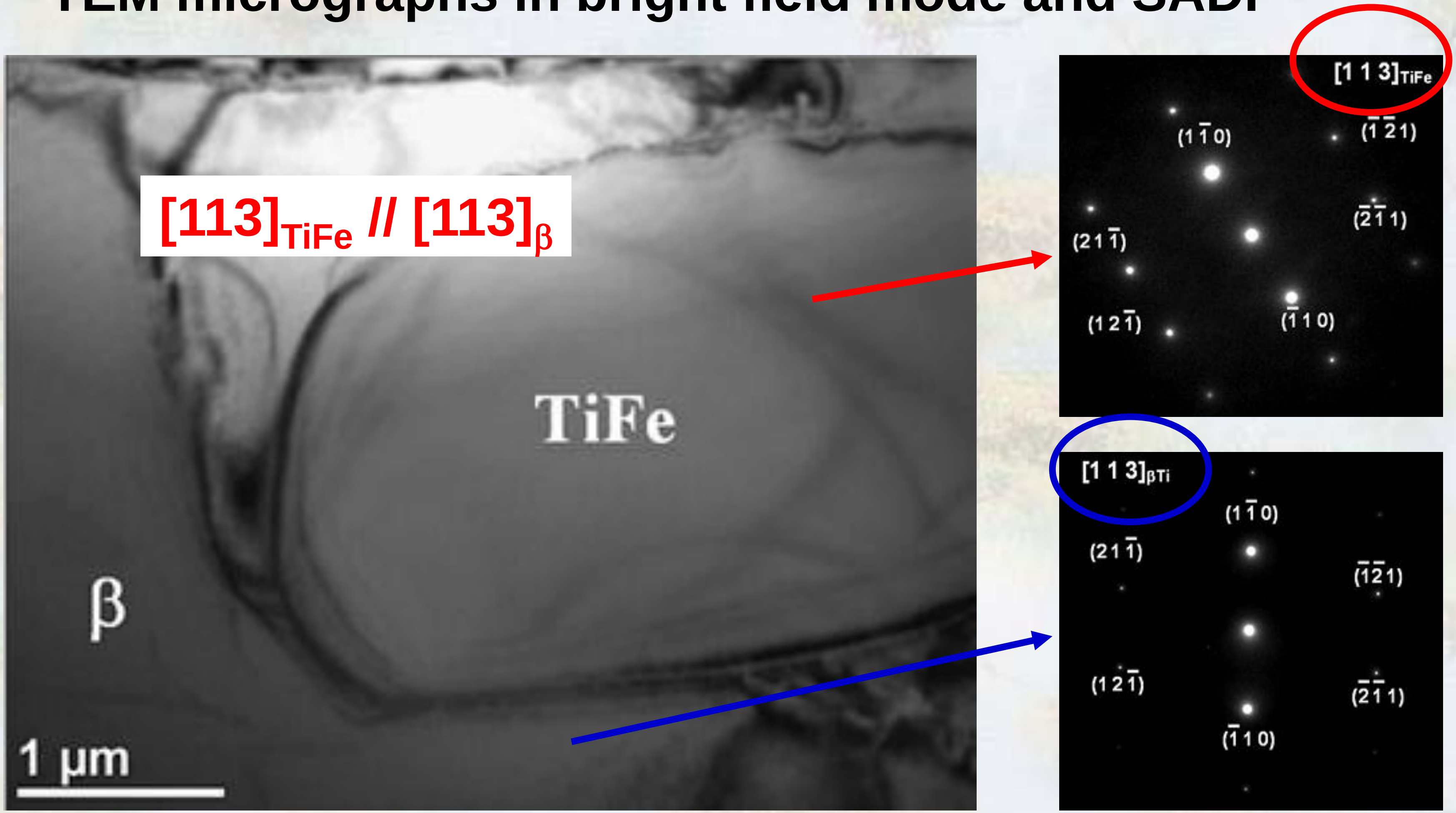
XRD Patterns

- **XRD patterns of Ti–Fe eutectic alloys in as-cast and directionally solidified (DS) conditions**



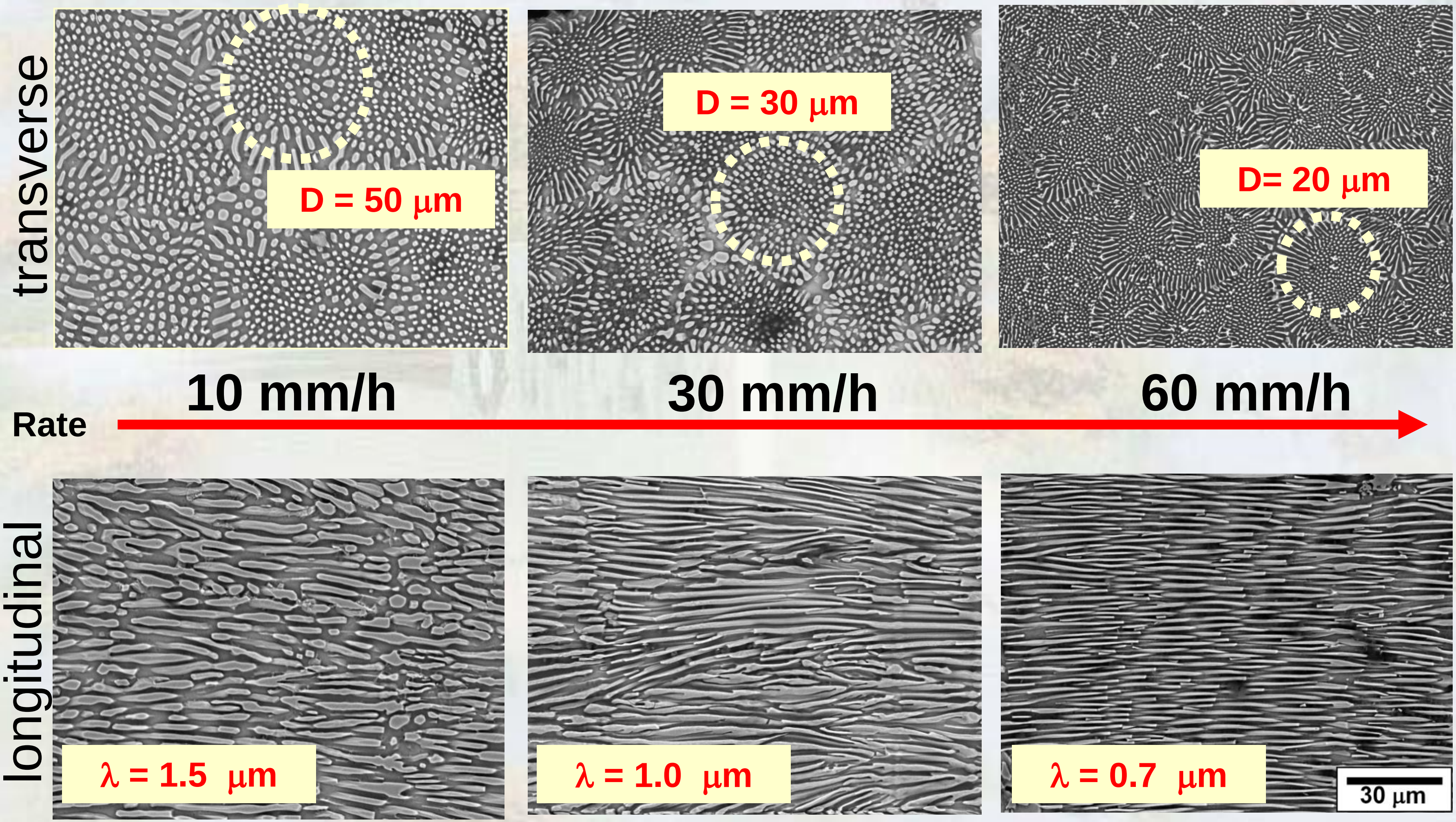
TEM Analysis

- **TEM micrographs in bright field mode and SADP**



Solidification Rate

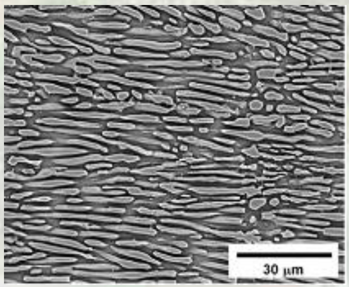
- **SEM micrographs showing transverse and longitudinal cross-sections at different rates**



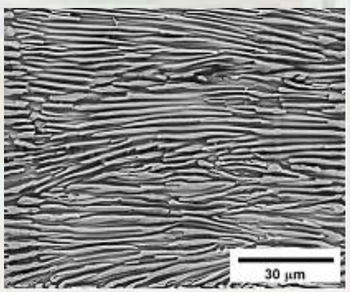
Solidification Rate

- **Relationship between the average interspace and the solidification rate of d.s. Ti-Fe eutectic alloy**

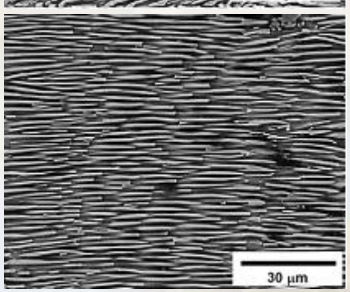
10 mm/h

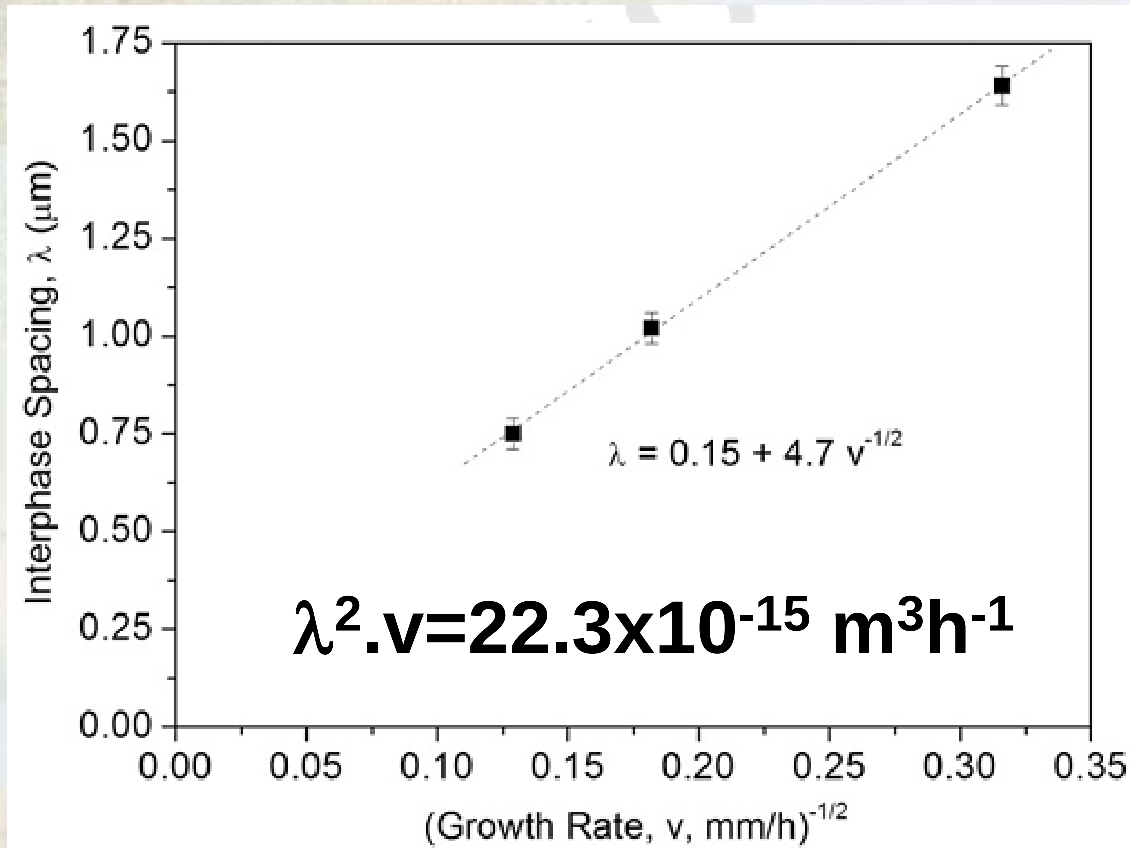


30 mm/h



60mm/h

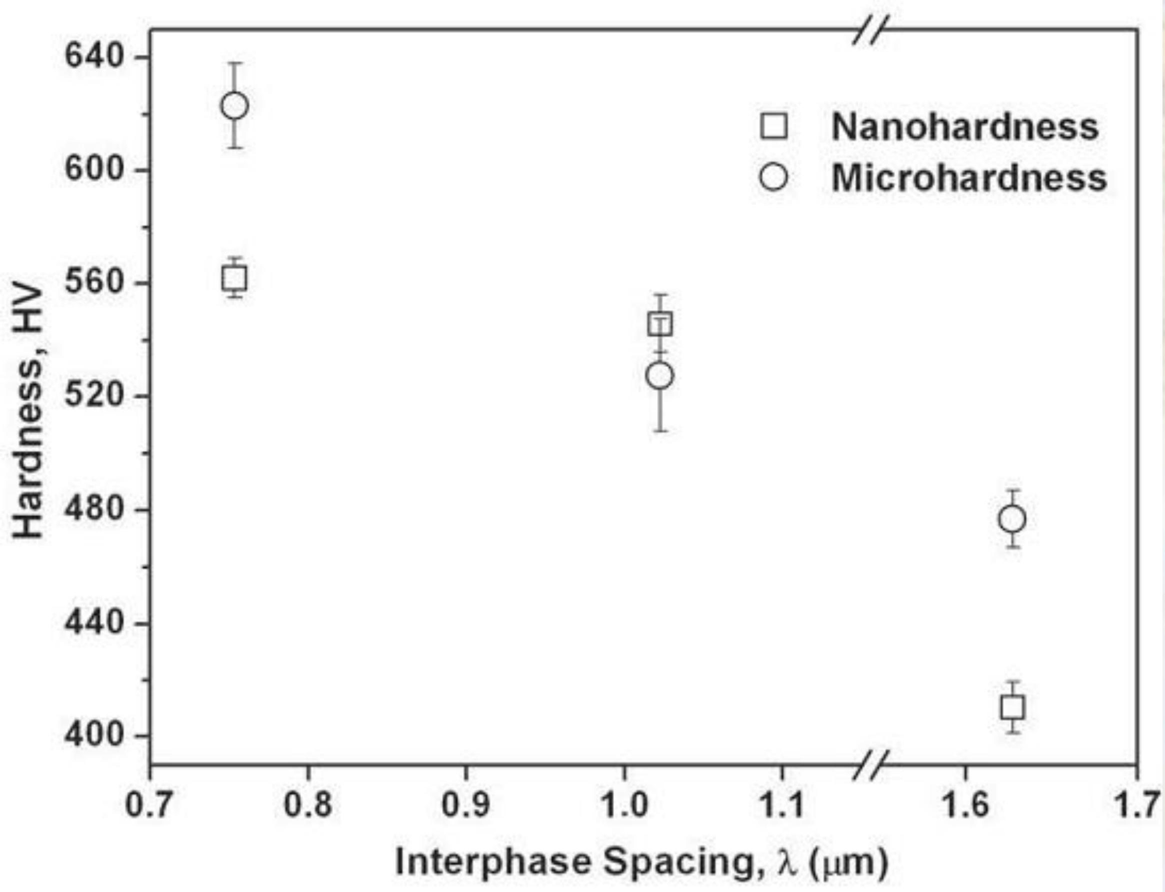




$$\lambda^2 . v = 22.3 \times 10^{-15}\ m^3 h^{-1}$$

Mechanical Tests

- **Evolution of hardness with interphase spacing versus Vickers microhardness (HVmicro) and nanohardness (HVnano).**



Nano-indentation: Three-sided Berkovich diamond indenter and applying a maximum load of 500 Mn:

E_E=110 to 177 GPa

E_{TiFe} = 137 GPa

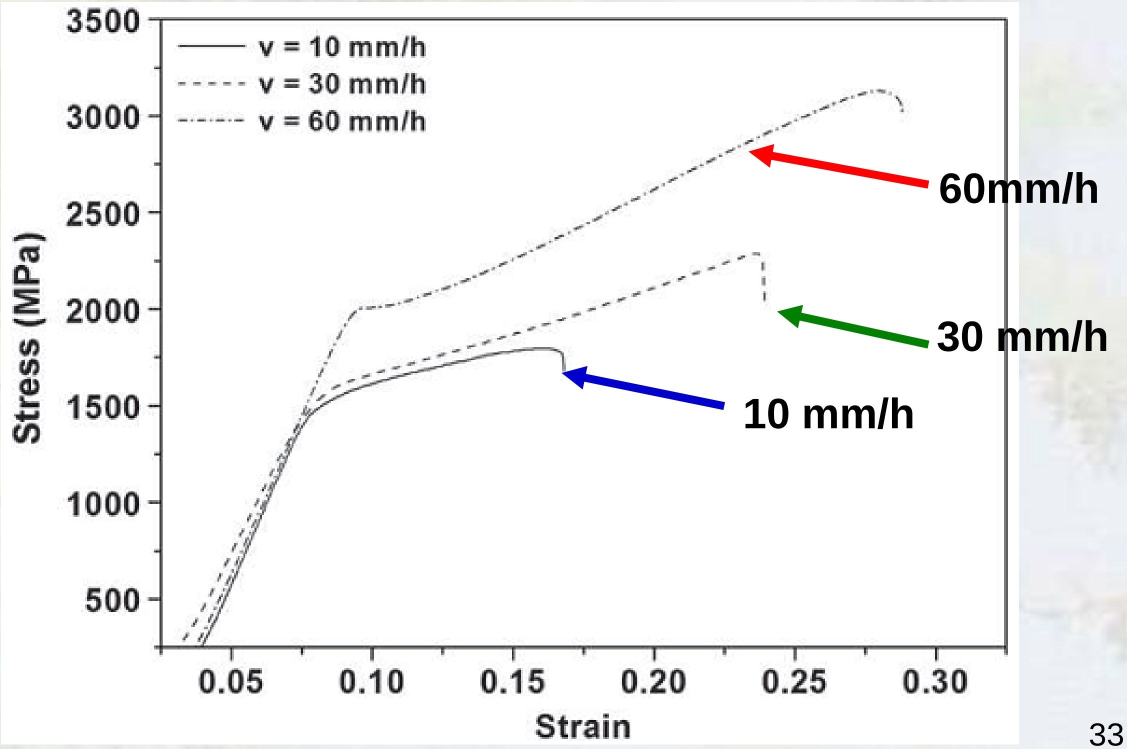
Mechanical Tests

- **Compressive mechanical properties**

λ (μm)	Ultimate stress σ_{max} (MPa)	Yield strength σ_y (MPa)	Yield strain ε_y (%)	Fracture strain ε_f (%)
0.7	3000±137	1926±69	11.7±1	25.2±2
1.0	2494±161	1790±124	8.5±1	23.0±1
1.5	1844±101	1644±72	8.1±1	21.6±1

Compression tests:

- **Strain rate of $8 \times 10^{-3} s^{-1}$**
- **Samples 4 mm high and 2 mm in diameter**



Conclusions

New experimental setup was applied to D.S. of Ti-Fe eutectic:

- **No oxygen contamination**
- **No evidence of oxygen rich phase**
- **Well aligned eutectic microstructure**
- **Eutectic transformation at 1080ºC**
- **TEM/SADP**

  → orientation relationship: $(113)_{\beta} \| (113)_{TiFe}$
- **$\lambda^2 v = 22.3 \times 10^{-15}$ m³/h**
- **E_E varies from 110 to 177 GPa**
- **σ_{UTS} varies from 1844 to 3000 MPa**
- **Ductility varies from 21.5 to 25.2 %**

Acknowledgments

- The State of São Paulo Research Foundation
- The Brazilian National Council for Scientific and Technological Development

for financial support

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