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19th International Conference on Internal Friction and Mechanical Spectroscopy

The Conference, originally planned as hybrid online and face-to-face event in Moscow (http://icifms19.ru), will take place as a virtual event based in Rome 27.06-01.07.2022

Structural transformation in Al-51 at.% Zn eutectoid alloy observed in internal friction

S. Belhas[1], C. Belamri[1], S. Taabache[1],
S.A.E. Boyer[2-3], F. Hamon[3], A. Rivière[3], M. Gerland[3]

[1] Hadj Lakhdar Batna1 University – PRIMALAB Laboratory – Material Sciences Faculty – Route de Biskra - 05000 Batna, Algeria

[2] MINES ParisTech, PSL - Research University, Centre for Material Forming, UMR CNRS 7635, 1 Rue Claude Daunesse, 06904 Sophia Antipolis, France

[3] PPRIME Institute, ISAE- ENSMA, UPR CNRS 3346, Department of Physics and Mechanics of Materials, 1 Av. Clément Ader, 86961 Futuroscope Chasseneuil Cedex, France

WHAT ABOUT Al-Zn STUDIES

- Zn-Al based alloys have been commercially accepted for many years, since the first casting alloy was developed at the New Jersey Zinc Company in 1922

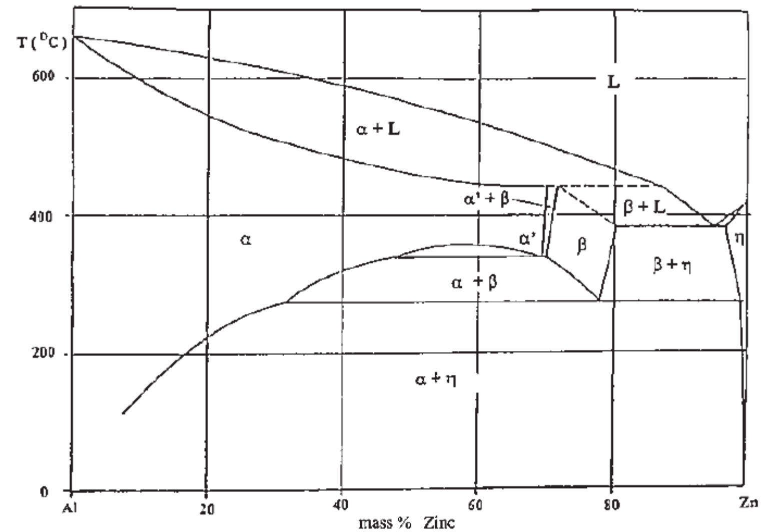
General Rule of Phase Decomposition in Zn-Al Based Alloy

Since the 1970's, systematic investigation of phase relationships in Zn-Al (ZA) based alloys

Studies began with the establishment of phase diagrams of alloy systems (Zn-Al binary, Zn-Al-Cu, Zn-Al-Si ternary and Zn-Al-Cu-Si quaternary)

Followed by studies of the phase transformations which occurred in solution-treated alloys

Phase diagram of Presnyakov et al. modified by Goldak and Parr has been adopted for representation of Zn-Al binary phase diagram [Zhu2004]

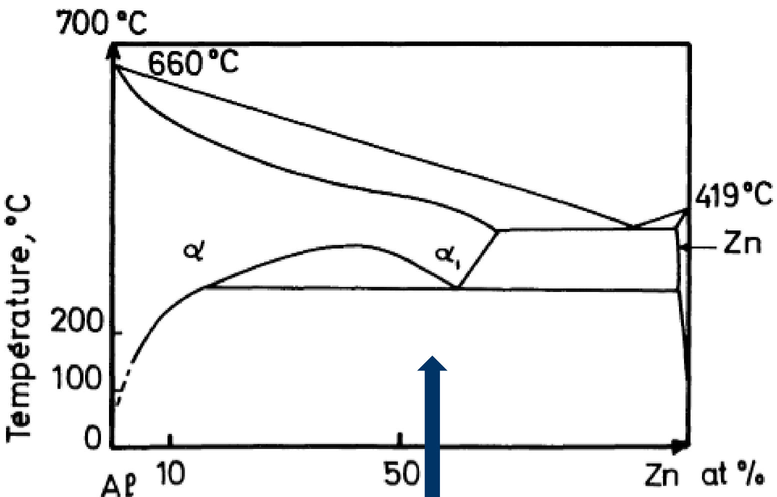


[Zhu2004] *Y.H. Zhu, 'General Rule of Phase Decomposition in Zn-Al Based Alloys (II) -On Effects of External Stresses on Phase Transformation-': Materials Transactions, 45(11) (2004) 3083-3097*

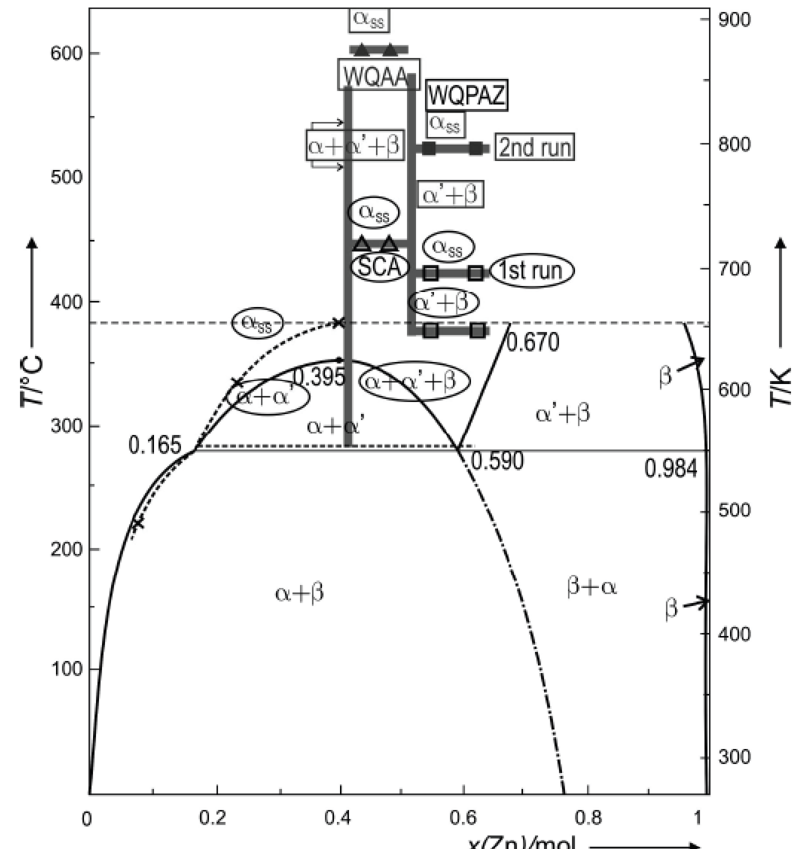
[Presnyyakovu1961] *A.A. Presnyyakov, Y.A. Goltban, V.C. Cherptyyakova: Russ. J. Phys. Chem. 35 (1961) 623-633*

WHAT ABOUT Al-Zn PHASES DIAGRAM

- Zn-Al phases diagram ‘light’ to ‘heavy’ information



Al-51 at.% Zn



[Dartyge1969] *E. Dartyge, J.M. Dartyge, M. Lambert, A. Guinier, ‘Étude de la résistivité d’une solution solide Al-Zn au cours de sa précipitation’: Journal de Physique, 30(1) (1969) 82-86*

[Željko2009] *S. Željko, P. Stanko, S. Goran, ‘Microstructure of Al-Zn and Zn-Al Alloys’: Croatica Chemical Acta 82(2) (2009) 405-420*

WHAT ABOUT Al-Zn STUDIES & microSTRUCTURES

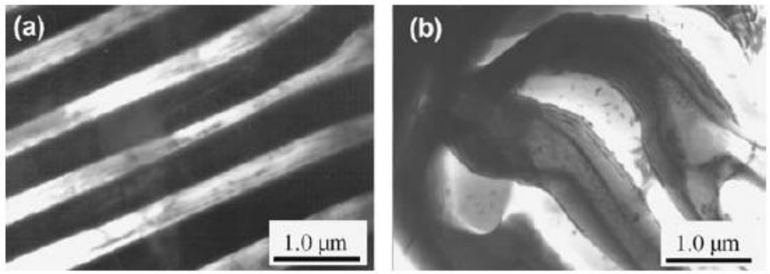
Among these materials, the eutectoid alloy Al 51%.at Zn

At the microstructural level, observations by different authors [Zhu1997] [Zhu2004] [Hirata1998] [Zhang2006] on the eutectoid alloy in particular showing that the configuration of the mixture of the two phases α and β is very complex, thermo and mechanical dependent

- Lamellar structures, deformed lamellar structures, globular structures, interconnected globular structures and clusters.

In addition, different aspects (chemical composition, phase evolution, etc.) & try to identify the transition from one structure to another [Zhu1997] [Hirata1998] [Zhu2004]

However, no statistical study established to indicate the proportion of each 'state'



TEM microstructures in Zn–Al eutectoid alloy after solution treatment at 360C-6hrs, followed by furnace cooling. β-(dark plate) and α-(bright plate) phases ; (a) Straight lath, and (b) curved lath

[Zhang2006] *M.-X. Zhang, P.M. Kelly, 'Understanding the crystallography of the eutectoid microstructure in a Zn-Al alloy using the edge-to-edge matching model': Scripta Materialia 55 (2006) 577-580*

[Hirata1998] *V.M.L. Hirata, M. Saucedo Munoz J.C. Rodriguez Hernandez Y.H. Zhu, 'Milling characteristics of extruded eutectoid Zn–Al alloy': Materials Science and Engineering A247 (1998) 8-14*

[Zhu1997] *Y.H. Zhu, V.M.L. Hirata, M.S. Munoz, 'Milling induced microstructural change in furnace cooled eutectoid Zn single bond Al alloy': Journal of Materials Processing Technology 63 (1997) 624-627*

Al-51 at.% Zn alloy eutectoid alloy: mechanical spectroscopy?

1. Introduction

State of the art, thermal behavior & structural transitions in Al-Zn alloys

- Isothermal Mechanical Spectroscopy *versus* Structure Analysis

2. Forced pendulum *versus* SEM analysis

At constant temperature, At low frequency

Activation parameters

Structure Analysis: **A**(lamellar) to **E**(globular)

3. Physical Analysis in Al-51 at.% Zn alloy

Stabilised structures & Spectroscopy spectra

4. Discussion/Conclusion

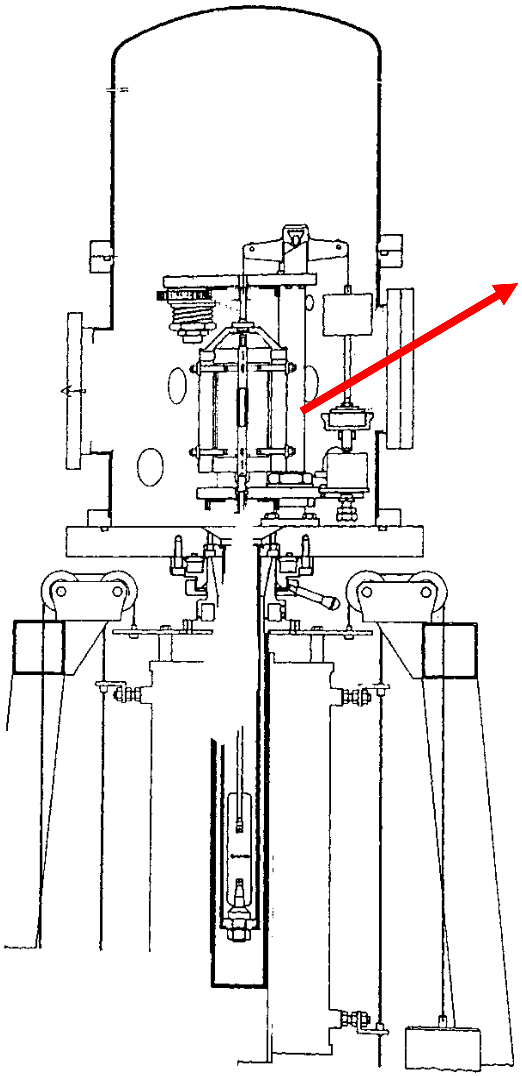
[Rivière2012] *A. Rivière, V. Pelosin, M. Gerland, 'Eutectoid Al-51at%Zn Alloy Studied by Isothermal Mechanical Spectroscopy': Solid State Phenomena 184 (2012) 167-172*

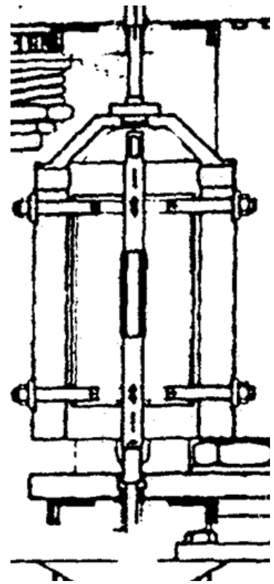
[Zhu1990] *X.F. Zhu, 'Stable damping associated with linear viscous motion of the interface in a multiphase Al-Zn alloy': Journal of Applied Physics 67 (1990) 7287 (1990)*

THE FORCED PENDULUM: Temperature & Low Frequency

4

• **Stress-Strain Dephasage Measurement**





The moving part is suspended by four very fine crossed ribbons to prevent any possibility of flexion

⇒ Only isothermal experiments

Characteristics for internal friction experiments:

- Frequency range: 10^{-5} Hz - 50 Hz
- Temperature range: 300 K - 1400 K
- Maximal strain: 2×10^{-7} - 5×10^{-6} - 2×10^{-5}
- Damping accuracy: 10^{-4}

[Rivière[2001]] *A. Rivière, in: R. Schaller, G. Fantozzi, G. Gremaud (Eds.): 'Mechanical Spectroscopy Q^{-1} 2001 with Applications to Materials Science', Trans Tech Publication Ltd., Switzerland, 2001, p. 635,*

Materials & Procedure: SEM analysis

Binary **Al-51 at.% Zn**, ***eutectoid alloy*** (Goodfellow Company)

- ✓ Let's fix and observe: ***Figs. 1 & 2*** with the general appearance on quenched sample (first treatment 653K) from 385 and 493K, 2hrs30min holding time for each $T_{annealing}$
- ✓ Presence of "greyish" zones more preponderant in 385K sample compare to 493K ; ***Fig.1*** lamellar structure predominates, ***Fig.2*** globular structure, which is dominant

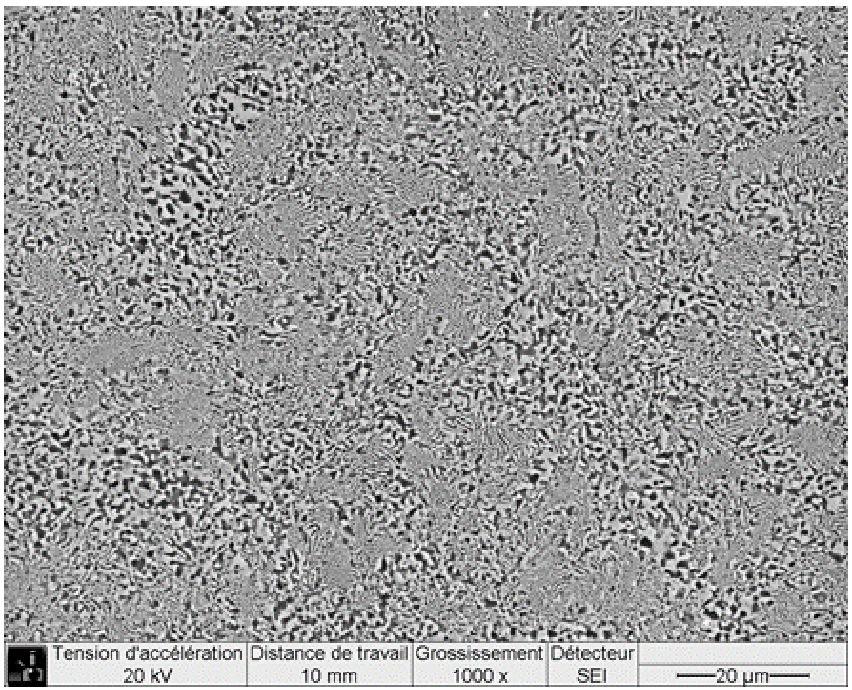


Fig.1 *Al-51% at. Zn, quenched from 385K*

SEM-FEG (JEOL 7000 Scanning Electron Microscopy-Field Emission Gun operating at 7 kV)

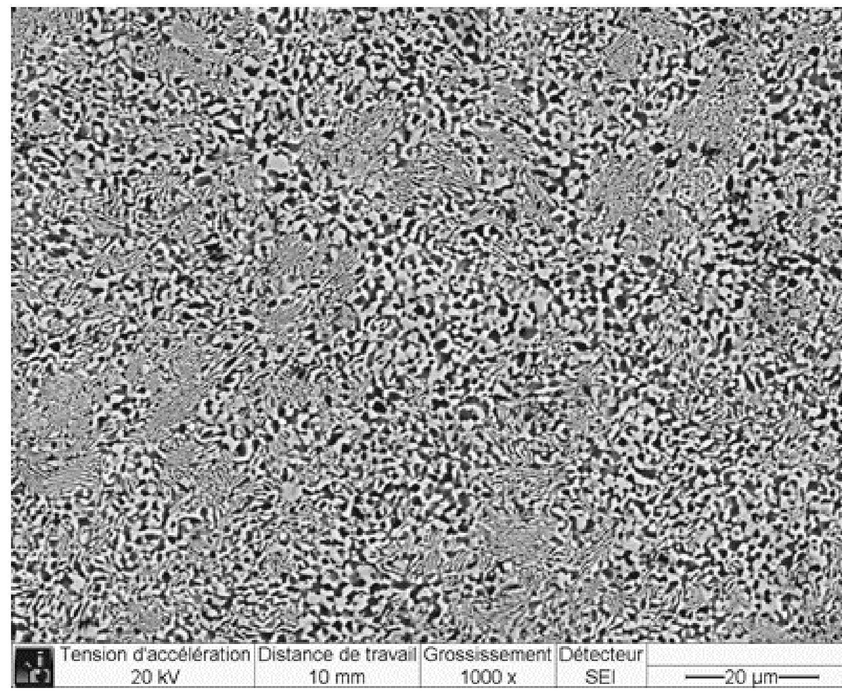


Fig.2 *Al-51% at. Zn, quenched from 493K*

Al-51 at.% Zn: SEM *vs. FDIF methodologies*

- Evolution of the two structures, SEM analyzed into small areas segmentation:

A, lamellar structure (greyish)

B, destruction of the lamellar structure and progressive evolution towards the globular one

C, formation of the globular structure

D & **E**, respectively, beginning and end of the globules process coalescence

- SEM size (1200 pixels in width) (900 pixels in height) divided into 885 square zones (3.95% of the total) of 35 pixels
- Procedure counting the number of each species (**A** to **E**) for SEM images for each T: average of all photos from the different observation areas of the same sample

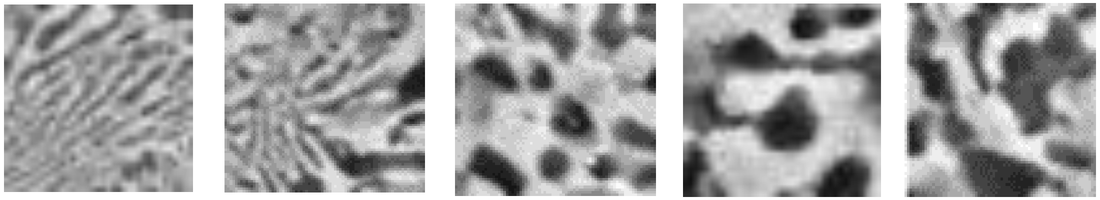


Fig.3 *Al-51% at. Zn,* successive elementary surfaces describing (**A** to **E**)

- SEM (385, 433 and 493K - 2hrs30min) chosen in T range of the internal friction peaks evolution

- ***FDIF***

Frequency dependent internal friction

- Annealing

(24hrs, vacuum 10^{-3}Pa)

- Temperatures steps

Atm to 540K

- Deformation

max. 5.10^{-6}

- Frequency

10^{-5}- 50Hz

SEM STRUCTURE ANALYSIS: Al-51 at.% Zn, 385K

- Statistically detection of 854 surfaces presented in the form of a histogram (***Fig.4***)
- **B**, corresponding to the destruction of the lamellar structure and its progressive transformation into a globular structure, is preponderant
- ➢ This change from **A** to **B** necessarily induces a change in the shape of the interface between α and β phases

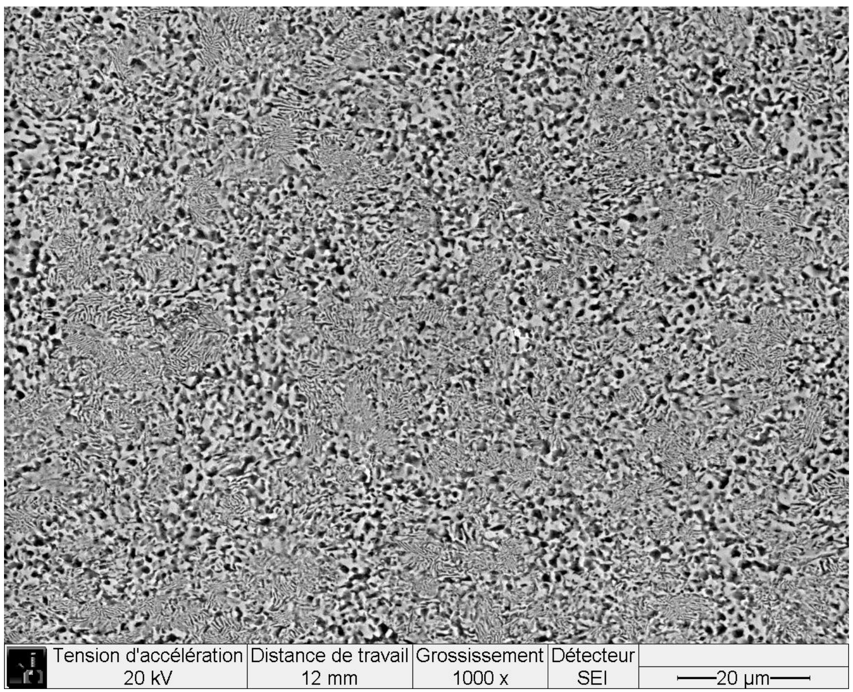


Fig.4 *Al-51% at. Zn, quenched from 385K*

Histogram *Al-51% at. Zn, 385K*

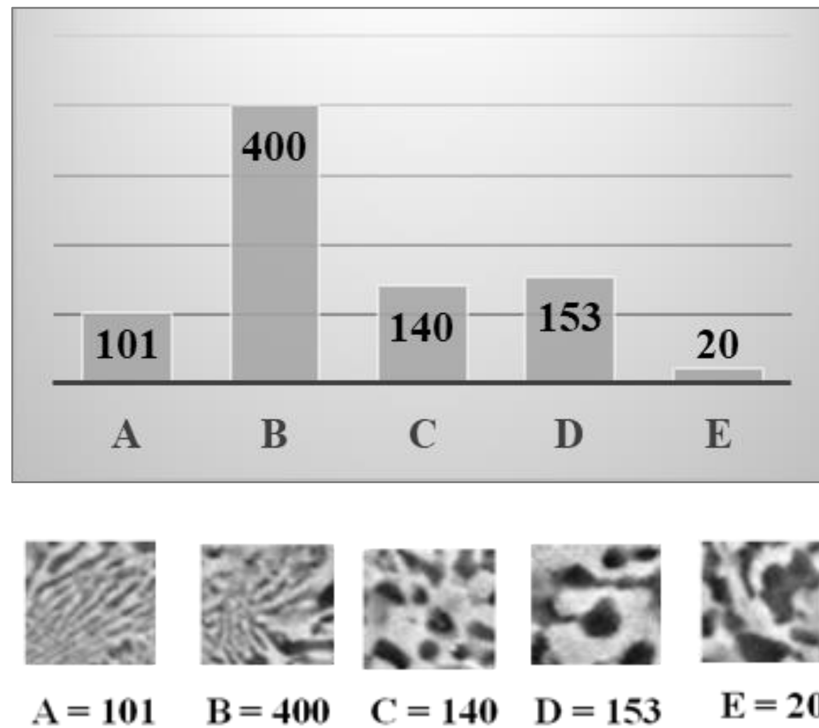


Fig.4 *Al-51% at. Zn, 385K: SEM analysis*

SEM STRUCTURE ANALYSIS: Al-51 at.% Zn, 433K

- Statistically detection of 794 surfaces, histogram (*Fig.5*)
- All the area A to E exist
- ➢ However, predominant areas B and D with practically an equal importance: correspond respectively to the destruction of the lamellar and globular structure

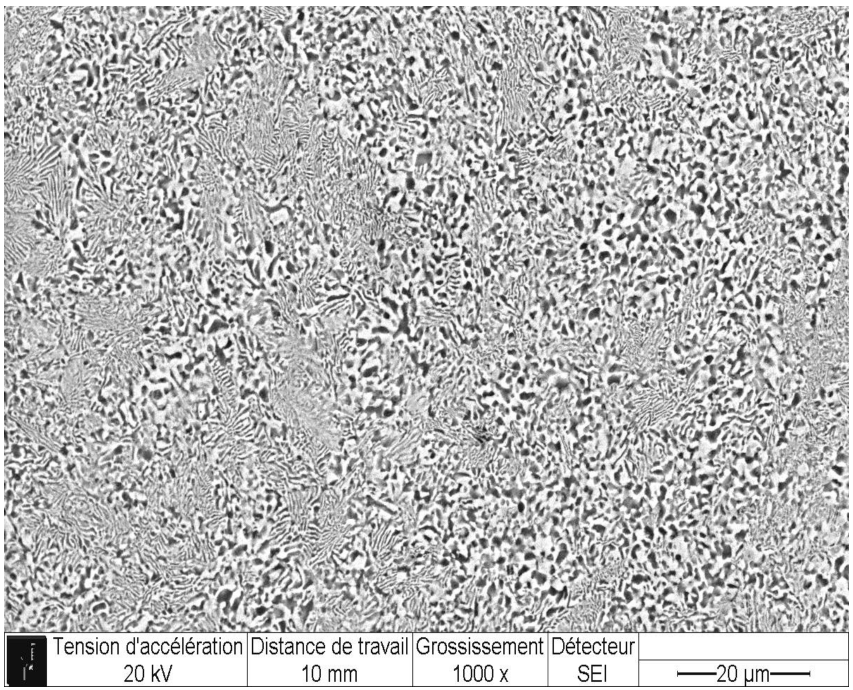


Fig.5 Al-51% at. Zn, quenched from 433K

Histogram Al-51% at. Zn, 433K

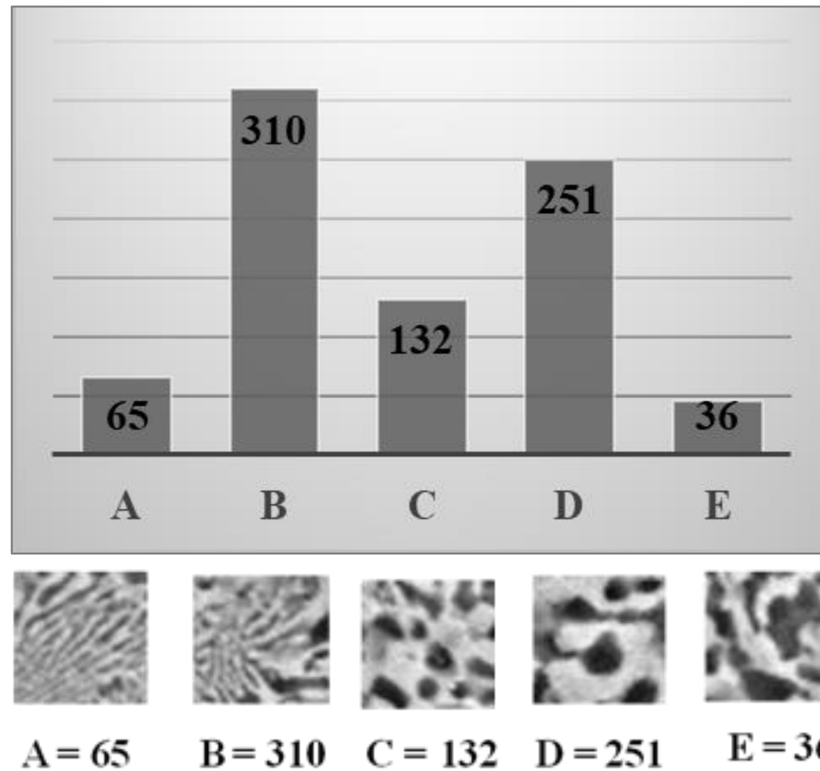


Fig.5 Al-51% at. Zn, 433K: SEM analysis

SEM STRUCTURE ANALYSIS: Al-51 at.% Zn, 493K

- Statistically detection of 832 surfaces presented in the form of a histogram (***Fig.6***)
- Significant increase in **D** and **E** surfaces categories, confirming the total transition from the lamellar structure to the coalescence of the globular structure
- ➢ Lamellar structure completely disappears, giving way to the coalescence of the globular structure

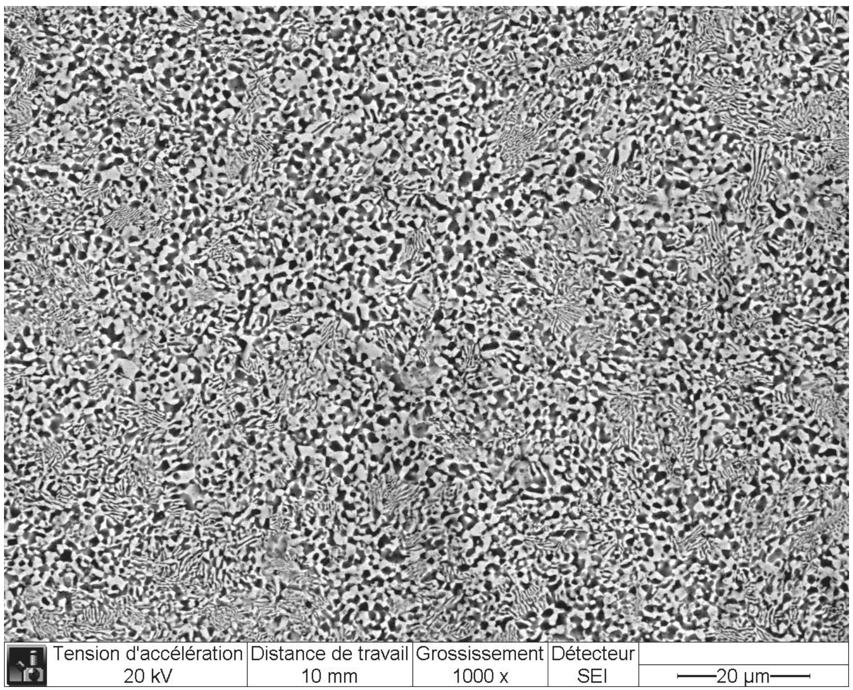
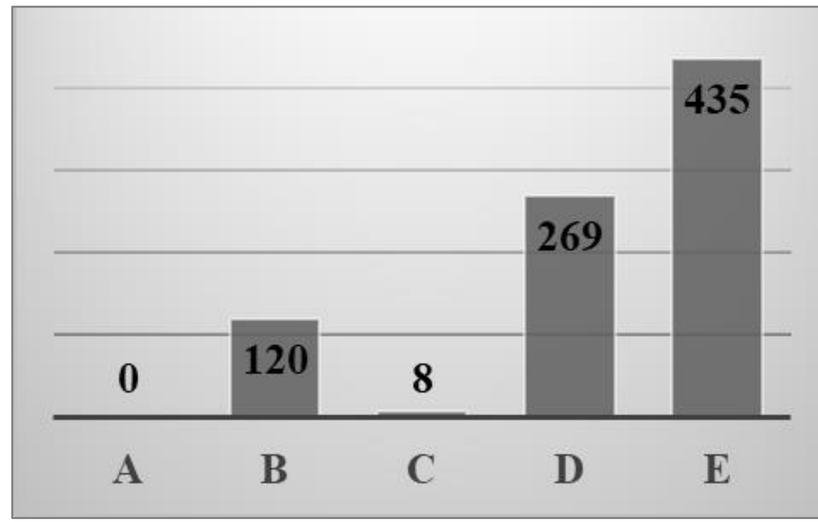


Fig.6 *Al-51% at. Zn, quenched from 493K*

Histogram *Al-51% at. Zn, 493K*



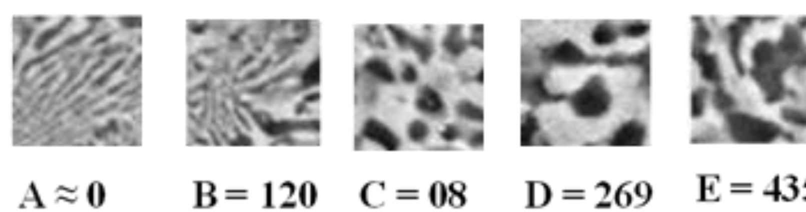


Fig.6 *Al-51% at. Zn, 493K: SEM analysis*

P1 and P2 closely related to the microstructural transformation?

Review: Frequency dependent Internal Friction
Spectra at temperature range 385-467K, during first heating

- Below the eutectoid transition temperature, the structure is a mixture of the two α-β phases

- IF (**Fig.7**) revelation of two relaxation peaks P1 and P2, where with increasing T>350K:
  - P1 shift to higher frequencies with a decrease in its amplitude
  - P2 appearance at higher frequencies and progressive increase

Behavior closely related to evolution of the microstructure obtained under the eutectoid transition ?

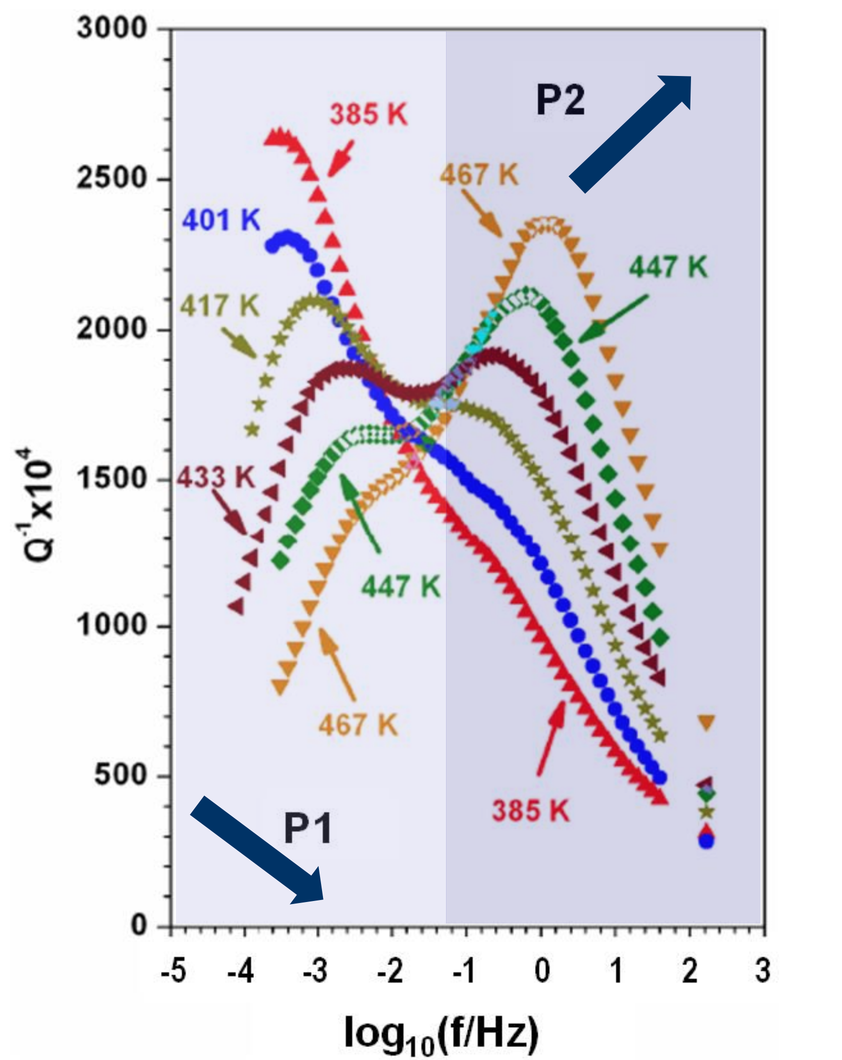


Fig.7 Low frequency Internal Friction Spectra, first heating at various measurement T

Al-51 at.% Zn PHYSICAL ANALYSIS: Activation energy

P1 and P2 thermally activated ★
Activation parameters during T decrease, 467, 527 and 544K
$H_Z = 0.75\text{eV}$, $\tau_0 = 10^{-7}$

- ➢ Close to Zhu, Al-60 at % Zn exp. in (290-390K) at (0.1 and 7Hz)

- [Zhu[1990]] observed damping because thermally induced atom diffusion across the interface between α and β phase grains during decreasing measurement T
- [Rivière[2012]] hypothesis to explain P1 and P2 co-existence, believes that the progressive ‘*P1 disappearance*’ and simultaneous ‘*P2 appearance & increase*’ could correspond to a change in the shape of the lamellar interface between α and β ? Together with atoms diffusion at the interface.

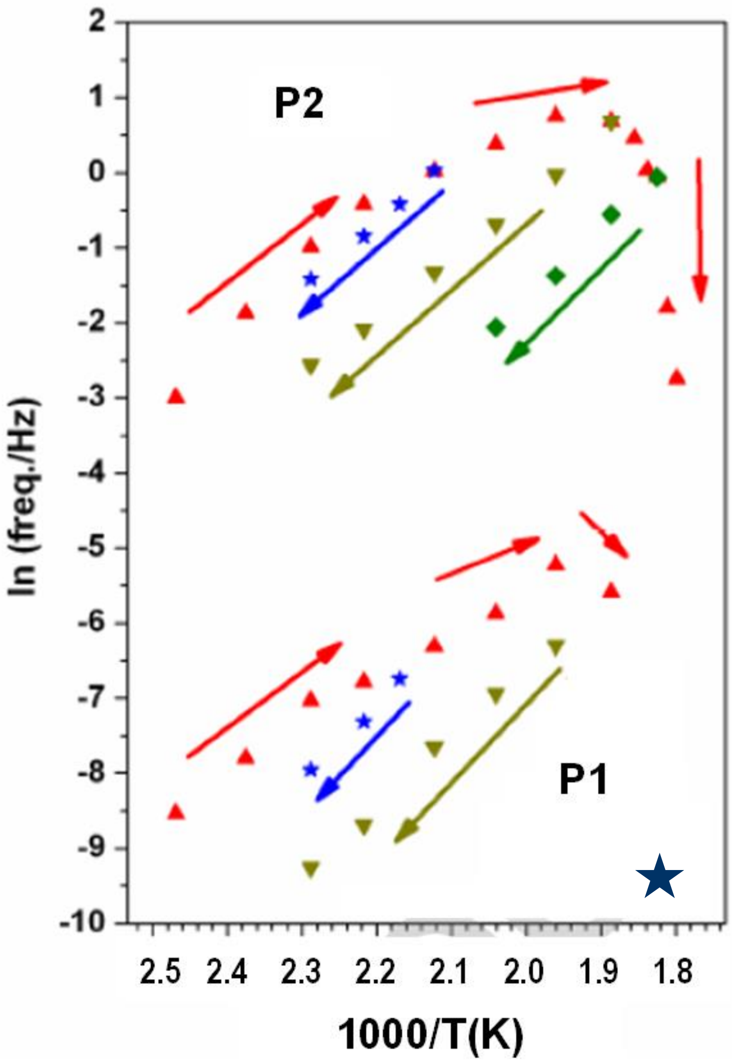


Fig.8 Napierian logarithm of frequency of peak maxima plotted *versus* inverse measurement T, first heating and after various annealing

Al-51 at.% Zn: FI *versus* Structures

Evolution of the microstructure obtained under the eutectoid transition

- SEM observations, to highlight the close link between the existence of the two peaks and the evolution of the microstructure

P1 FI-SEM-385K

- B preponderance, progressive lamellar structure destruction

Going in same direction as P1 evolution, whose relaxation strength gradually decreases with T increase

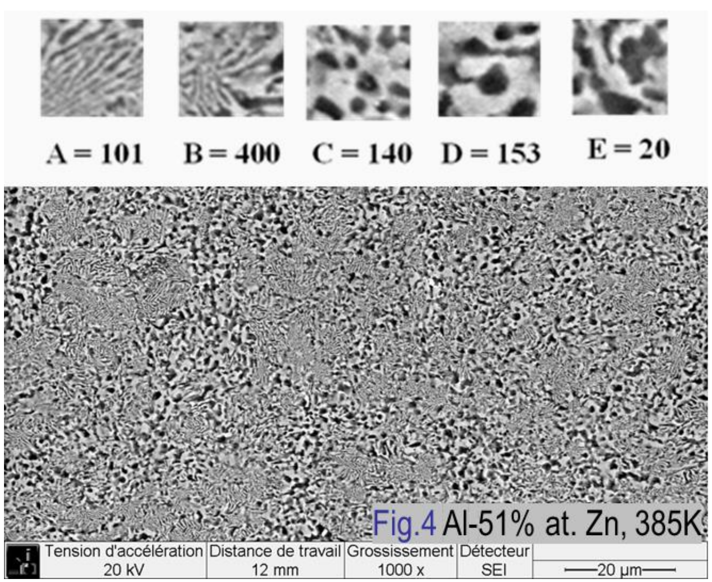
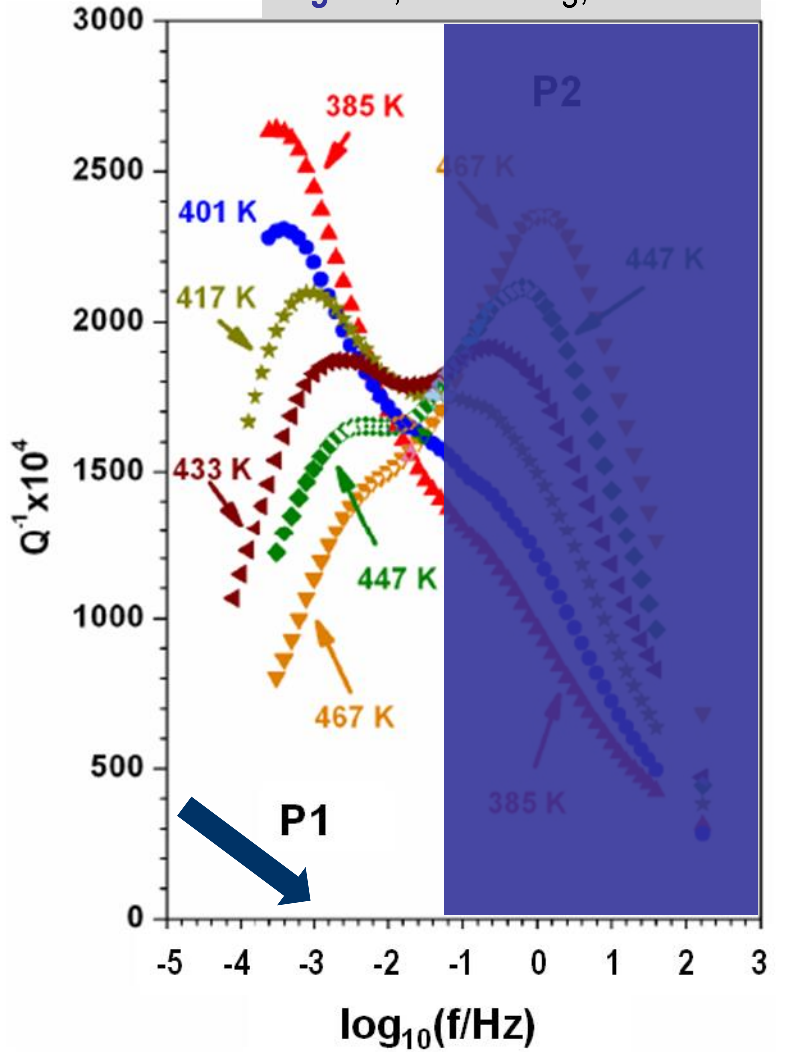


Fig.4 Al-51% at. Zn, 385K

Fig.7 IF, first heating, various T



Al-51 at.% Zn: FI *versus* Structures

P1 FI-385K / Confirmation during FI-SEM-433K

- SEM B and D in equal importance: respectively gradual destruction of lamellar to globular structure and beginning of coalescence of the globular structure

- *Agreement with P1 and P2 relaxation strengths, amplitude of the peaks practically equal

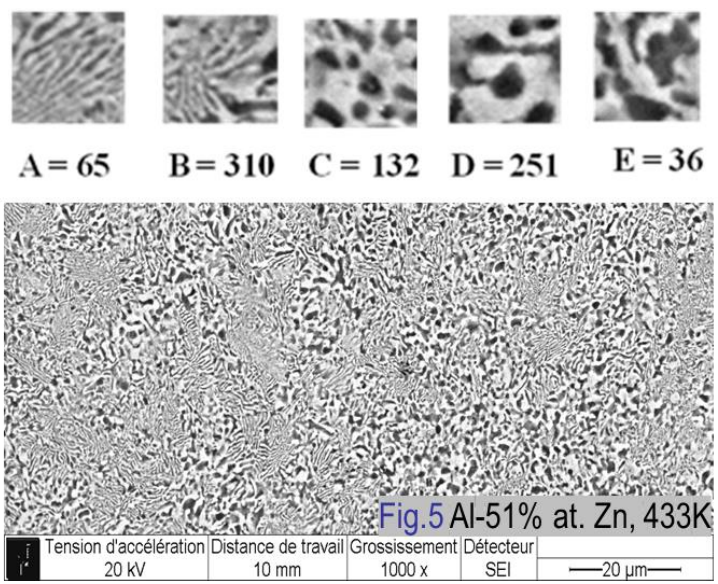
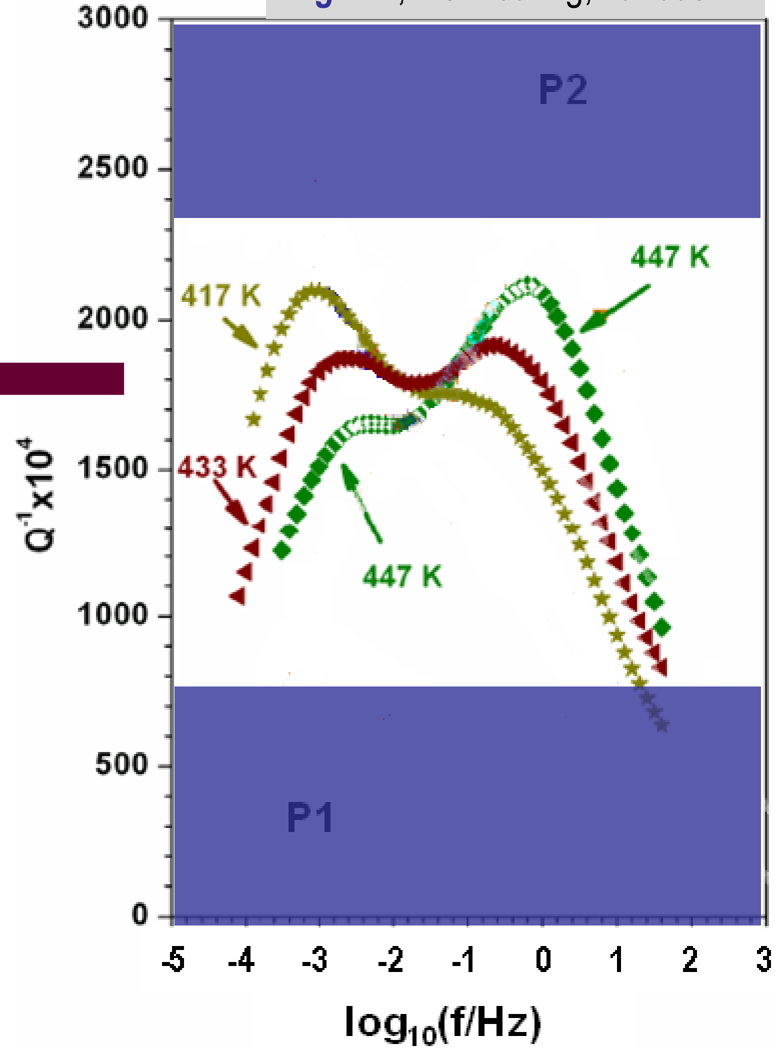


Fig.5 Al-51% at. Zn, 433K

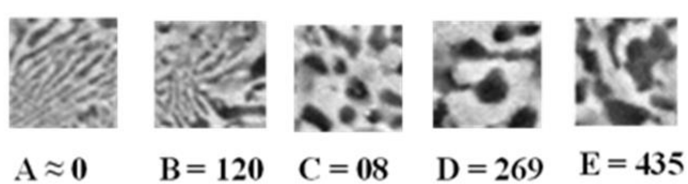
Fig.7 IF, first heating, various T



Al-51 at.% Zn: FI *versus* Structures

P2 FI-467K & SEM-493K

- D and E, corresponding to destruction of the globular structure predominate
- Development of P2 to the detriment of P1
- Behaviour globally reflects continuous *change in shape of interface* between α and β phases with their shape evolution ; *Zhu[1990] explains by the thermally induced atomic diffusion across the interface between the α and β phase grains*



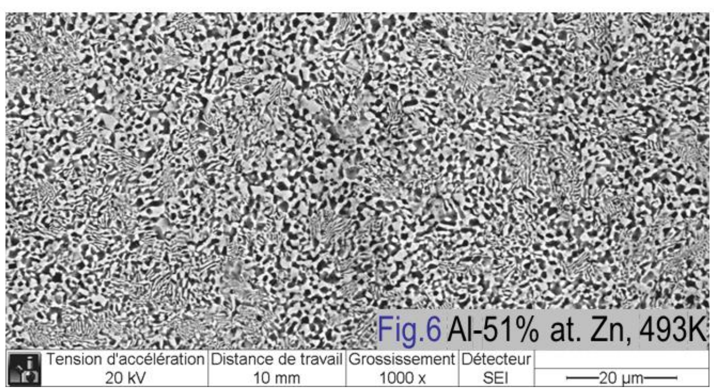
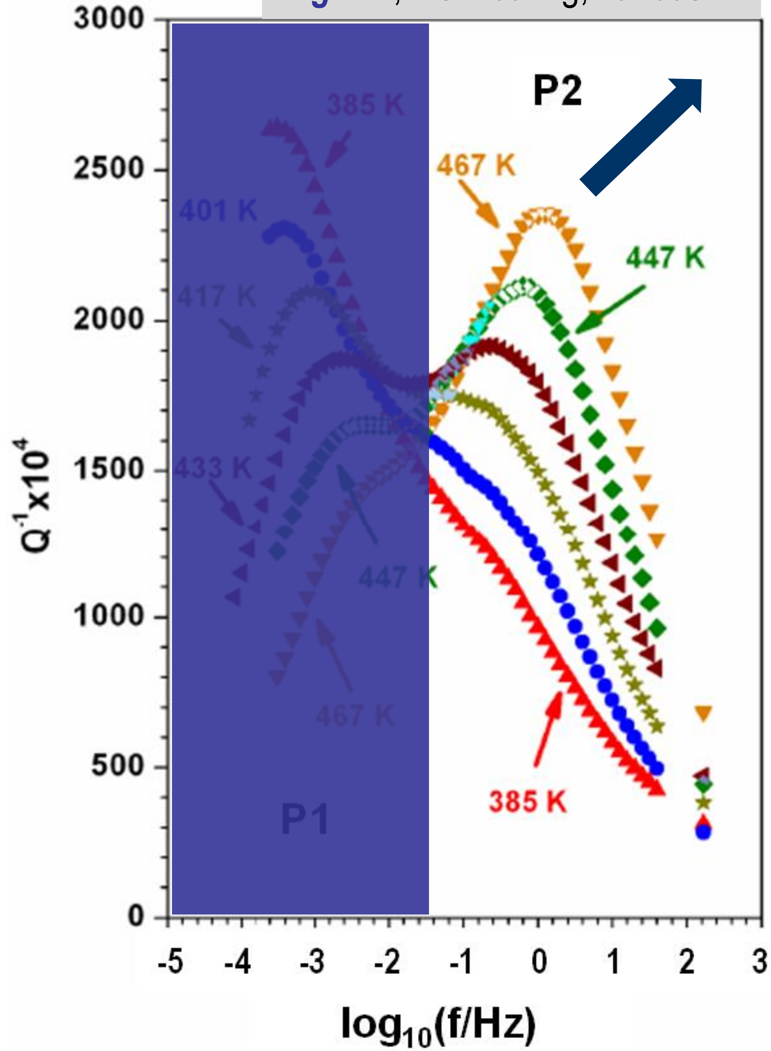


Fig.6 Al-51% at. Zn, 493K

Fig.7 IF, first heating, various T

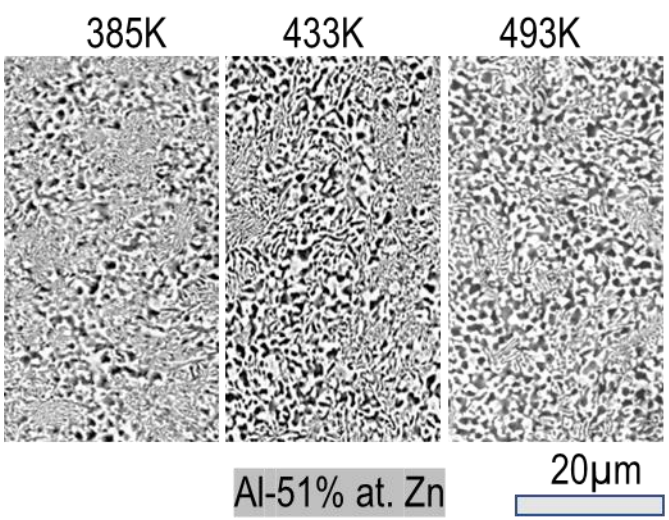


Al-51 at.% Zn: FI *versus* Structures

- **Arrhenius** (*Fig.8*)

Relaxation parameters seems to prove that we are in the presence of the same mechanism, thermal activation energy H is equal to 0.75eV (70kJ/mol) for both P1 and P2 with different microstructures

Thermally induced atoms diffusion across the α-β interface: indeed, fewer atoms involved in the elementary mechanism



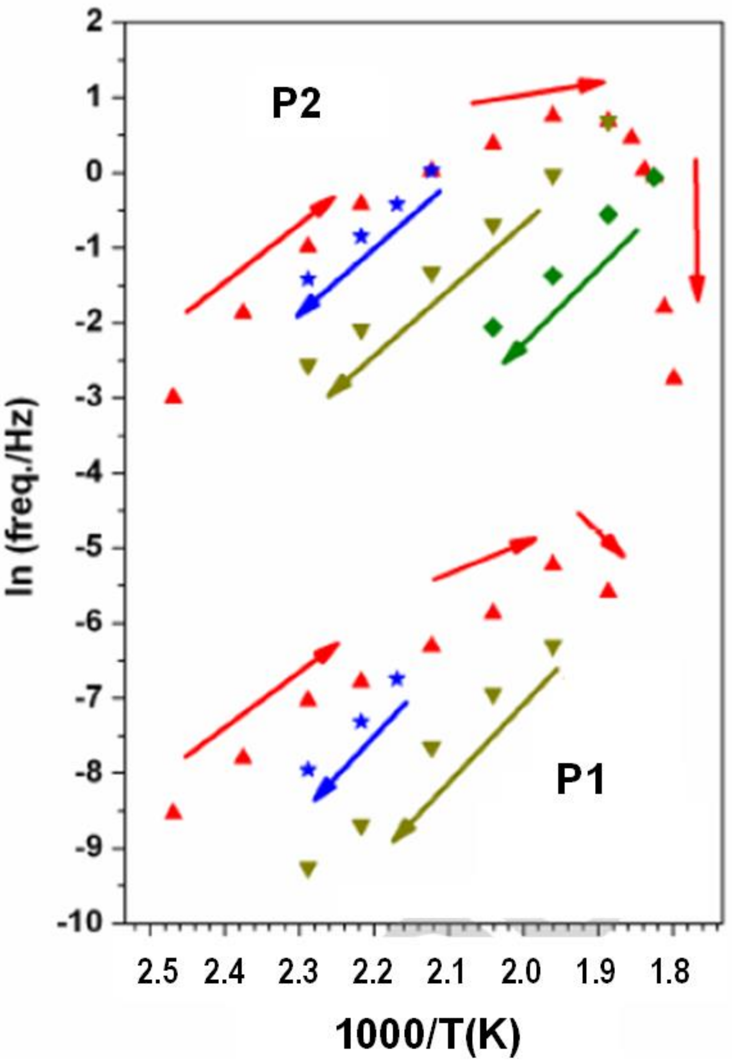


Fig.8 Napierian logarithm of frequency of peak maxima plotted *versus* inverse measurement T, first heating and after various annealing

CONCLUSION: ANALYSIS on Al-51 %.at Zn & Eutectoid

I. Al-51 at.% Zn alloy shows a total transformation of the eutectoid mixture at this %

- Ideal for studying the evolution of the alpha and beta phases as a function of temperature

II. Correlation between microstructure evolution (SEM) and isothermal mechanical spectroscopy (IMS)

- **IMS** carried out over wide frequency range (10^{-5}- 50 Hz) *vs.* T_{amb} to 540 K
- Upon heating, *two peaks* P1 and P2 below the eutectoid transition T_{eut} (550 K)
- Both thermally-activated ; P1 decreases and disappears with increasing T, while P2 appears and increases continuously until the eutectoid transition temperature

(P1 essentially, 383K) (*Coexistence P1/P2 same amplitude, 433K)* (P2 essentially, 467K)

- **SEM** after quenches at (385, 433 and 493 K - holding time of 2hrs30min)

P1 peak associated to lamellar structure whose destruction leads to the collapse of P1

P2 peak associated to coalescence of globular structure and as this coalescence the amplitude of this peak becomes more important

➢ Associated with thermally induced diffusion of atoms across the α-β interface ; microstructure from a lamellar form of the β-phase that gradually transforms according to the areas A and B until the globular form

III. SEM highlighting transformation mechanisms by specifying structure change and collapse

THANK YOU FOR YOUR KIND ATTENTION

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[1] Hadj Lakhdar Batna1 University – PRIMALAB Laboratory – Material Sciences Faculty – Route de Biskra - 05000 Batna, Algeria

[2] MINES ParisTech, PSL - Research University, Centre for Material Forming, UMR CNRS 7635, 1 Rue Claude Daunesse, 06904 Sophia Antipolis, France

[3] PPRIME Institute, ISAE- ENSMA, UPR CNRS 3346, Department of Physics and Mechanics of Materials, 1 Av. Clément Ader, 86961 Futuroscope Chasseneuil Cedex, France