Recrystallisation Kinetics of Plain Carbon Steels Containing Dilute Nb Additions

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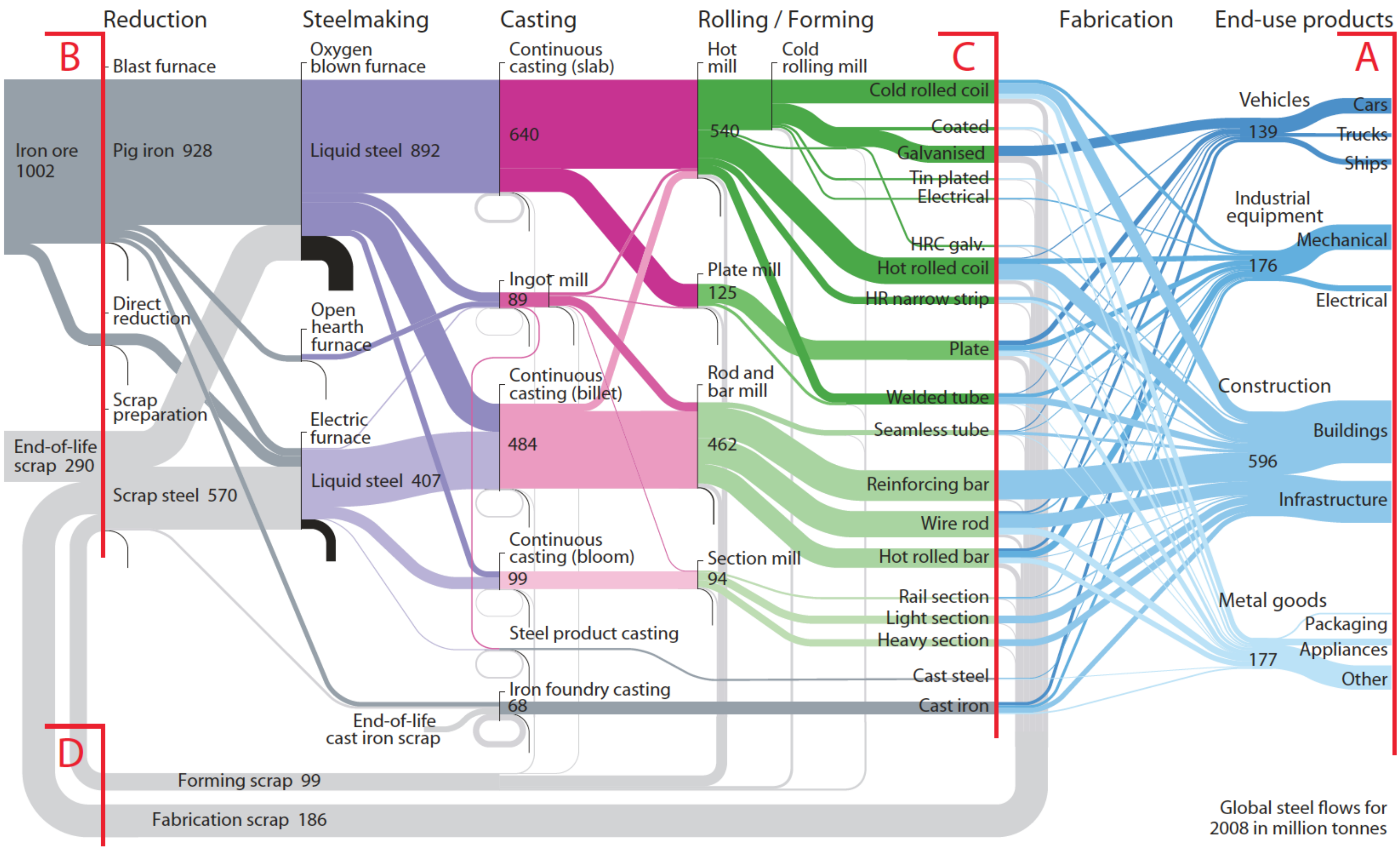
Map of global steel flow

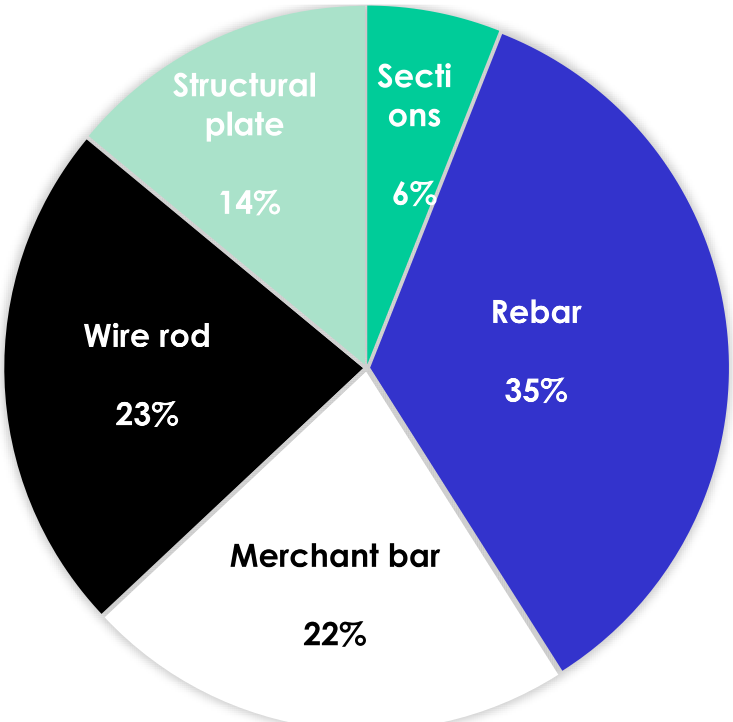


Source: Cullen,J.M., Allwood, J.M. and Bambach, M. (2012). Mapping the global flow of steel: from steelmaking to end- use goods, Environmental Science and Technology 46(24), 13048-13055.

Opportunity

2011 Global Steel Production, 1.500 billion

Structural Steels consists 500 million



Opportunity for Nb microalloying structural steel long products

Challenges

Very limited research in long steel products on recrystallisation and precipitate interaction

↓

Limited solubility of Nb in austenite for high carbon steels

↓

Rolling mill priorities
High speed, strain rates, temperature, times

Construction – complicated supply chain
Power lies with whom ?

↓

No generation shifts 2G/3G structural steels against automotive or oil & gas sector

Source: Steve Jansto, Nb-bearing construction steel and global application trends, Value added structural steel symposium, 2012, Singapore

Aims & Goals of Research

Aim: To investigate the effect of dilute Nb (<0.020 wt %) additions on recrystallisation behaviour of plain carbon steels (0.20 & 0.80 wt % C).

GOALS

- Study the effect of processing variables i.e. strain, deformation temperature & interpass time on static recrystallisation kinetics of steel grades.
- Determine the recrystallisation-stop ($T_{5\%}$) & limit ($T_{95\%}$) temperatures as function of a strain.
- Determine the driving (F_{RXN}) & retarding forces (F_{PIN}) for recrystallisation and correlate with $T_{5\%}$ & $T_{95\%}$.
- Investigate the effect of dilute Nb additions on mechanical properties of steel grades.

Research Methodology:

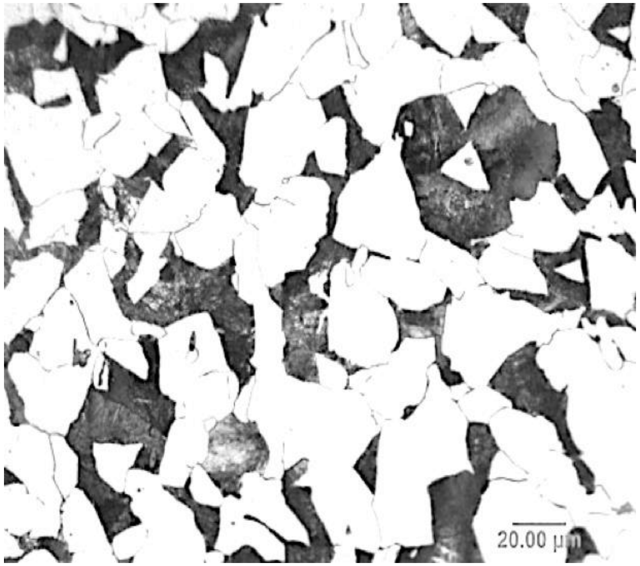
1. Plane Strain Compression (PSC) Testing, Fraction Softening Studies
2. Quantitative Metallography, OM and TEM
3. Modelling and Simulation

Chemical Composition

wt %

Steel Grades	C	Si	Mn	P	S	N	Nb
Base/C20	0.20	0.20	1.03	0.018	0.008	0.005	0.0
C20Nb05	0.20	0.19	1.03	0.018	0.008	0.005	0.004
C20Nb10	0.20	0.19	1.01	0.015	0.007	0.007	0.007
C20Nb20	0.20	0.19	1.01	0.015	0.007	0.008	0.017

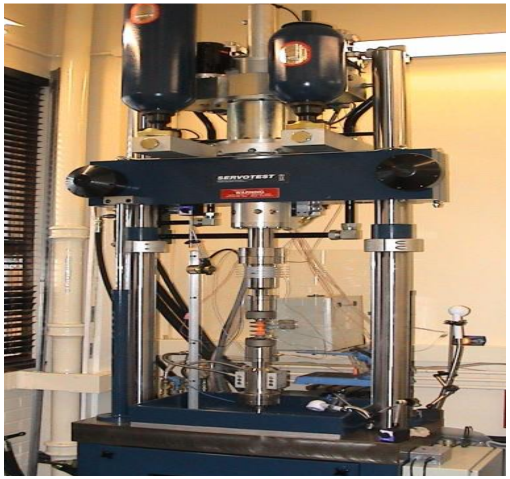
Laboratory Scale Heats
25 mm (T) x 105 mm (W) x 900 mm (L)



Avg. area fraction of pearlite: 31 ± 2 %
Vickers Hardness 164 ± 4 HV/1

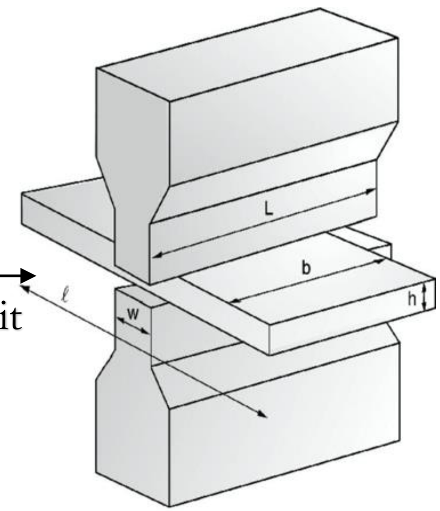
REX Simulation Techniques

Torsion - ASP

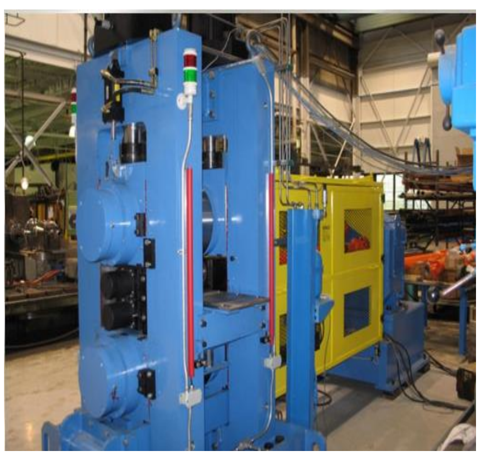


PSC - TMC

PSC Advantage



FENN Rev. HRM



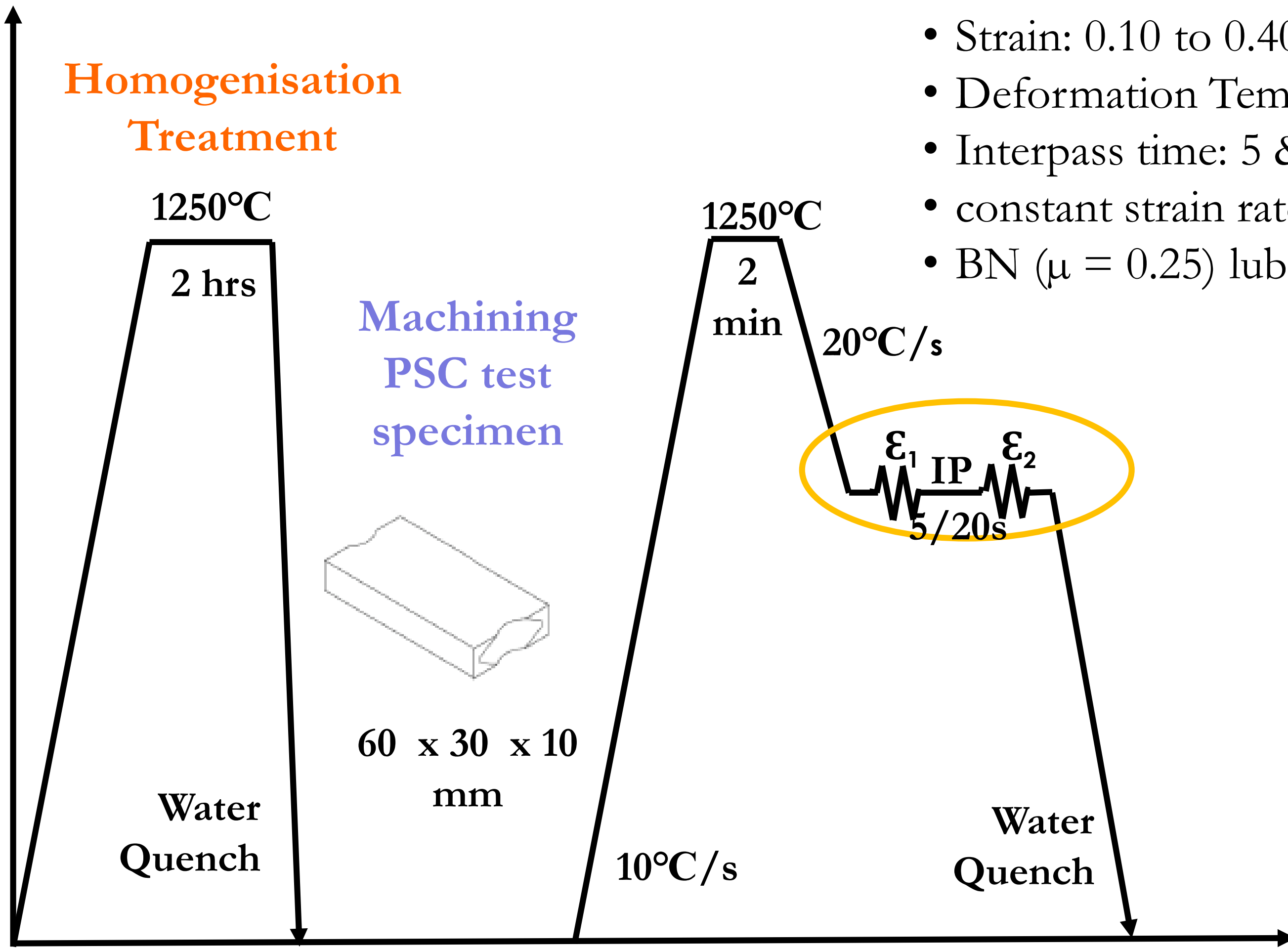
Advanced Thermal Treatment unit

Close to industrial condition & Large metallography area

Experimental Procedure

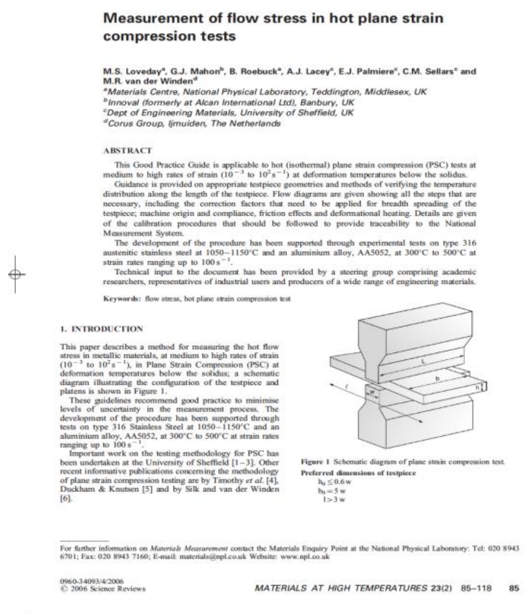
Double-hit simulation

- Strain: 0.10 to 0.40
- Deformation Temp: 850 - 1050°C
- Interpass time: 5 & 20s
- constant strain rate: $15s^{-1}$)
- BN (μ = 0.25) lubricant



Thermal Profile of Experiments

Flow Stress and Microstructure Analysis



Measurement of flow stress in hot plane strain compression tests

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[d]*Corus Group, Ijmuiden, The Netherlands*

ABSTRACT

This Good Practice Guide is applicable to hot (isothermal) plane strain compression (PSC) tests at medium to high rates of strain (10^{-3} to $10^{2}\,s^{-1}$) at deformation temperatures below the solidus.

Guidance is provided on appropriate testpiece geometries and methods of verifying the temperature distribution along the length of the testpiece. Flow diagrams are given showing all the steps that are necessary, including the correction factors that need to be applied for breadth spreading of the testpiece; machine origin and compliance, friction effects and deformational heating. Details are given of the calibration procedures that should be followed to provide traceability to the National Measurement System.

The development of the procedure has been supported through experimental tests on type 316 austenitic stainless steel at 1050–1150°C and an aluminium alloy, AA5052, at 300°C to 500°C at strain rates ranging up to $100\,s^{-1}$.

Technical input to the document has been provided by a steering group comprising academic researchers, representatives of industrial users and producers of a wide range of engineering materials.

Keywords: flow stress, hot plane strain compression test

1. INTRODUCTION

This paper describes a method for measuring the hot flow stress in metallic materials, at medium to high rates of strain (10^{-3} to $10^{2}\,s^{-1}$), in Plane Strain Compression (PSC) at deformation temperatures below the solidus; a schematic diagram illustrating the configuration of the testpiece and platens is shown in Figure 1.

These guidelines recommend good practice to minimise levels of uncertainty in the measurement process. The development of the procedure has been supported through tests on type 316 Stainless Steel at 1050–1150°C and an aluminium alloy, AA5052, at 300°C to 500°C at strain rates ranging up to $100\,s^{-1}$.

Important work on the testing methodology for PSC has been undertaken at the University of Sheffield [1–3]. Other recent informative publications concerning the methodology of plane strain compression testing are by Timothy *et al.* [4], Duckham & Knutsen [5] and by Silk and van der Winden [6].

Figure 1 Schematic diagram of plane strain compression test.

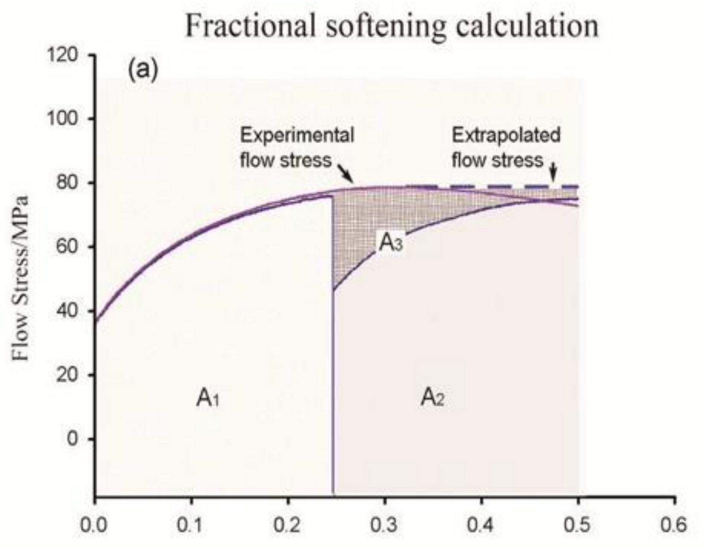
Preferred dimensions of testpiece

$h_0 \leq 0.6\,w$
$b_0 = 5\,w$
$l > 3\,w$

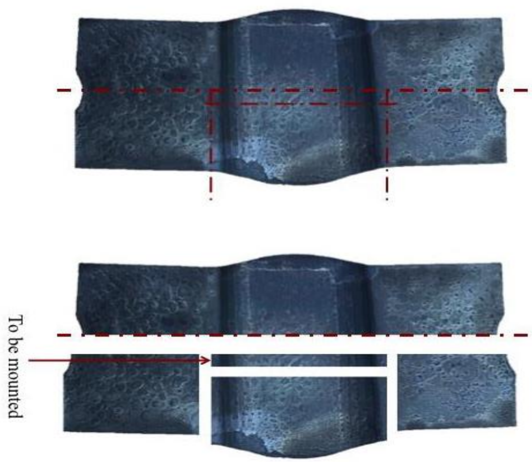
For further information on *Materials Measurement* contact the Materials Enquiry Point at the National Physical Laboratory: Tel: 020 8943 6701; Fax: 020 8943 7160; E-mail: materials@npl.co.uk Website: www.npl.co.uk

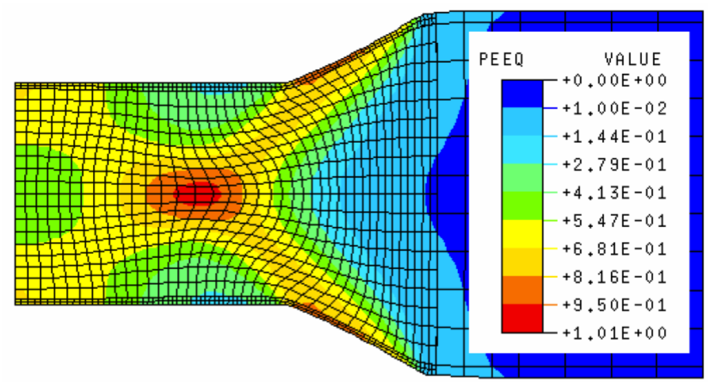
0960-34093/4/2006

MATERIALS AT HIGH TEMPERATURES **23**(2) 85–118 **85**



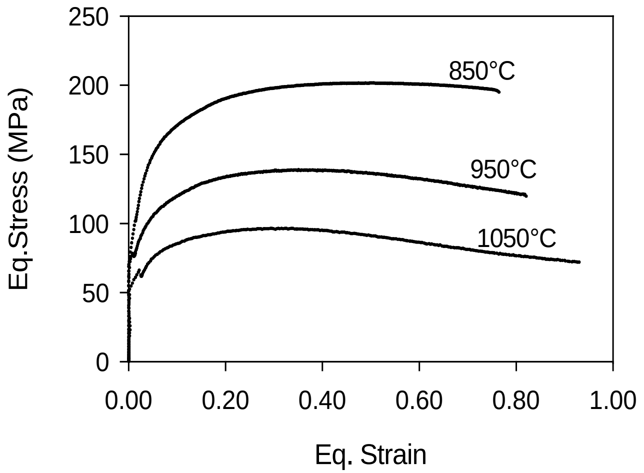
$$X_A = \frac{A_3 - A_2}{A_3 - A_1}$$



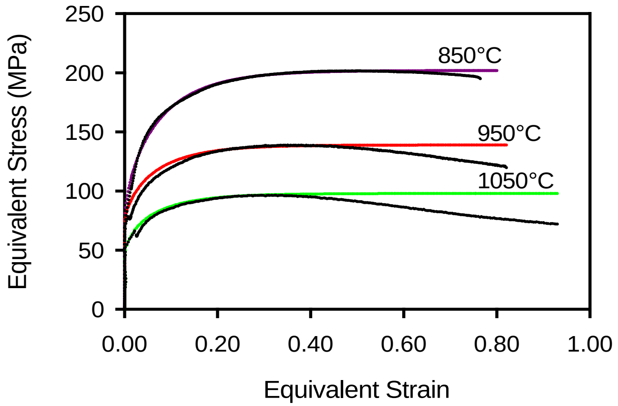


Equivalent Plastic Strain

Base – Single Heat Tests (SHT)

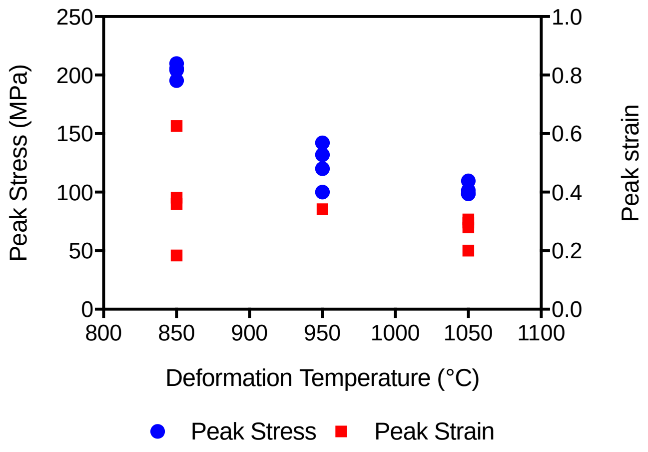


(a) Eq. stress-strain curve for SHT

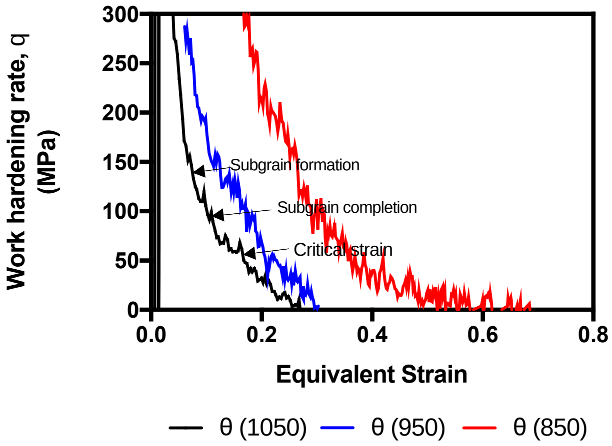


$$\sigma = \sigma_0 + (\sigma_{ss(e)} - \sigma_0)[(1 - \exp(-\varepsilon/\varepsilon_r)]^{1/2}$$

(b) Constitutive Equation Modelling

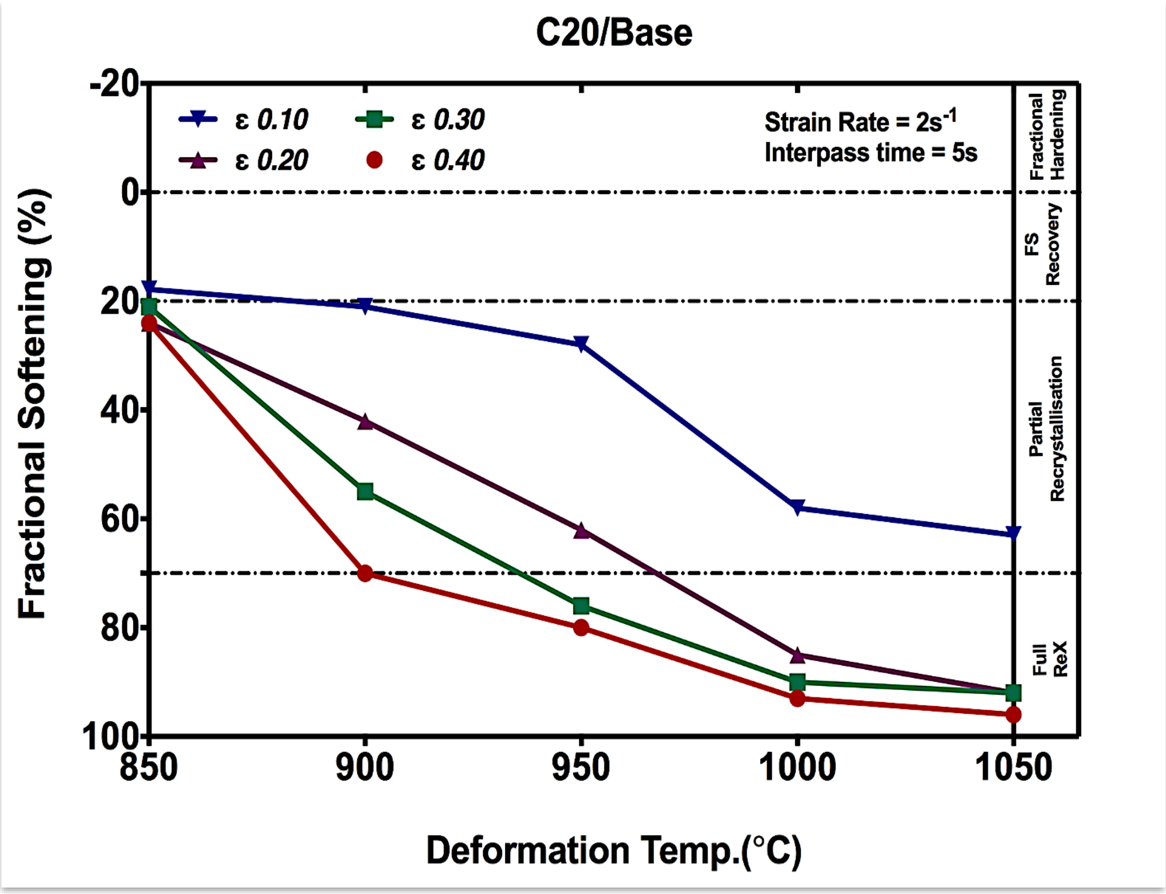


(c) DReX Analysis



(d) Critical Strain Analysis

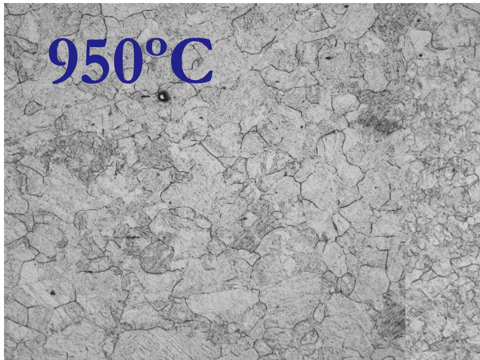
Softening fraction as a function of deformation temperature



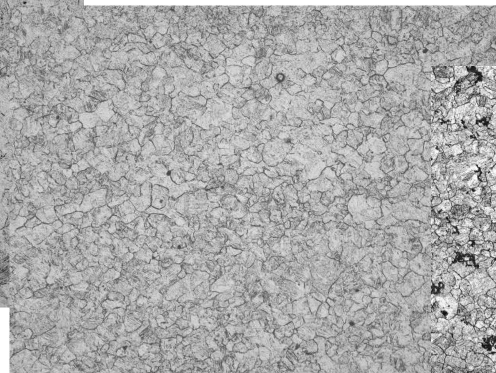
Inter-pass time: 5s

Note : All tests were performed at constant stain rate of 2 s^{-1}

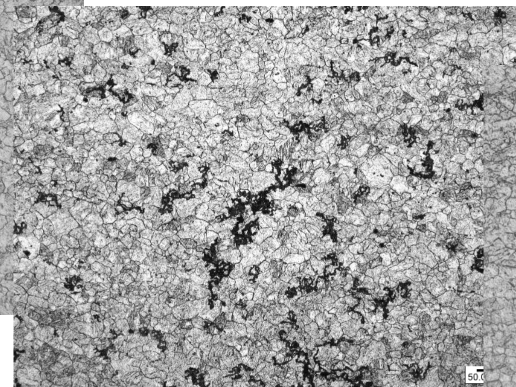
Double Hit Tests - IP 5s



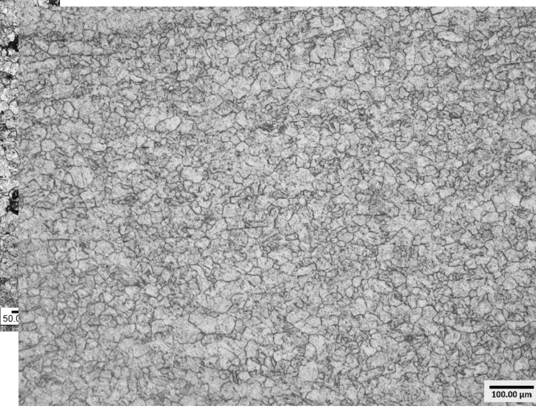
(a) Ɛ 0.10



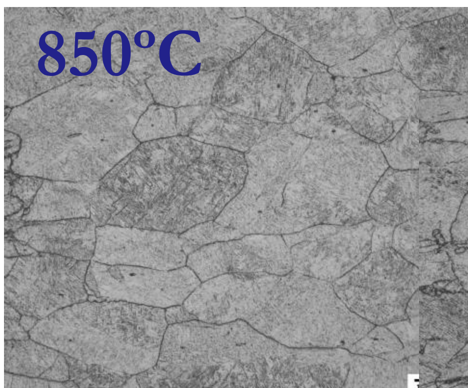
(b) Ɛ 0.20



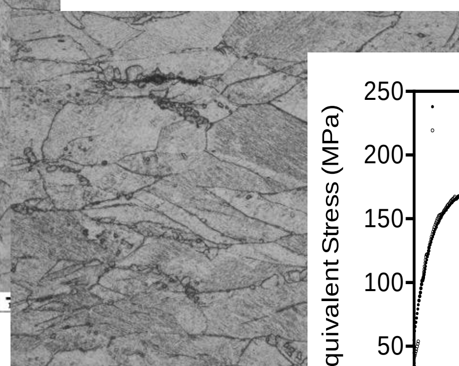
(c) Ɛ 0.30



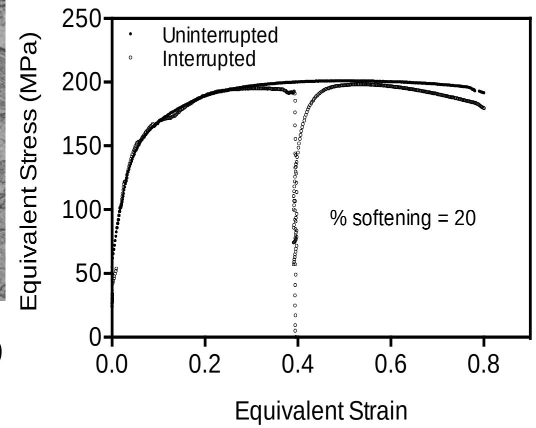
(d) Ɛ 0.40

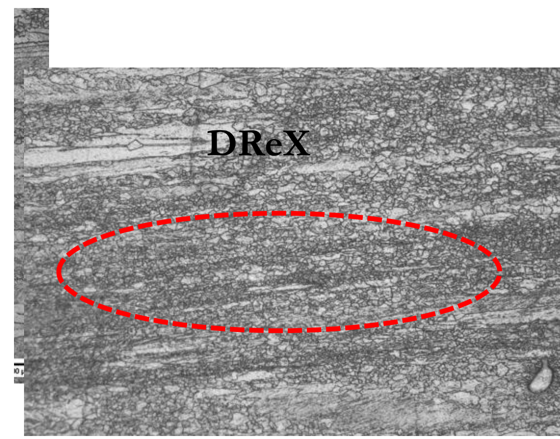


(a) Ɛ 0.10



(b) Ɛ 0.20

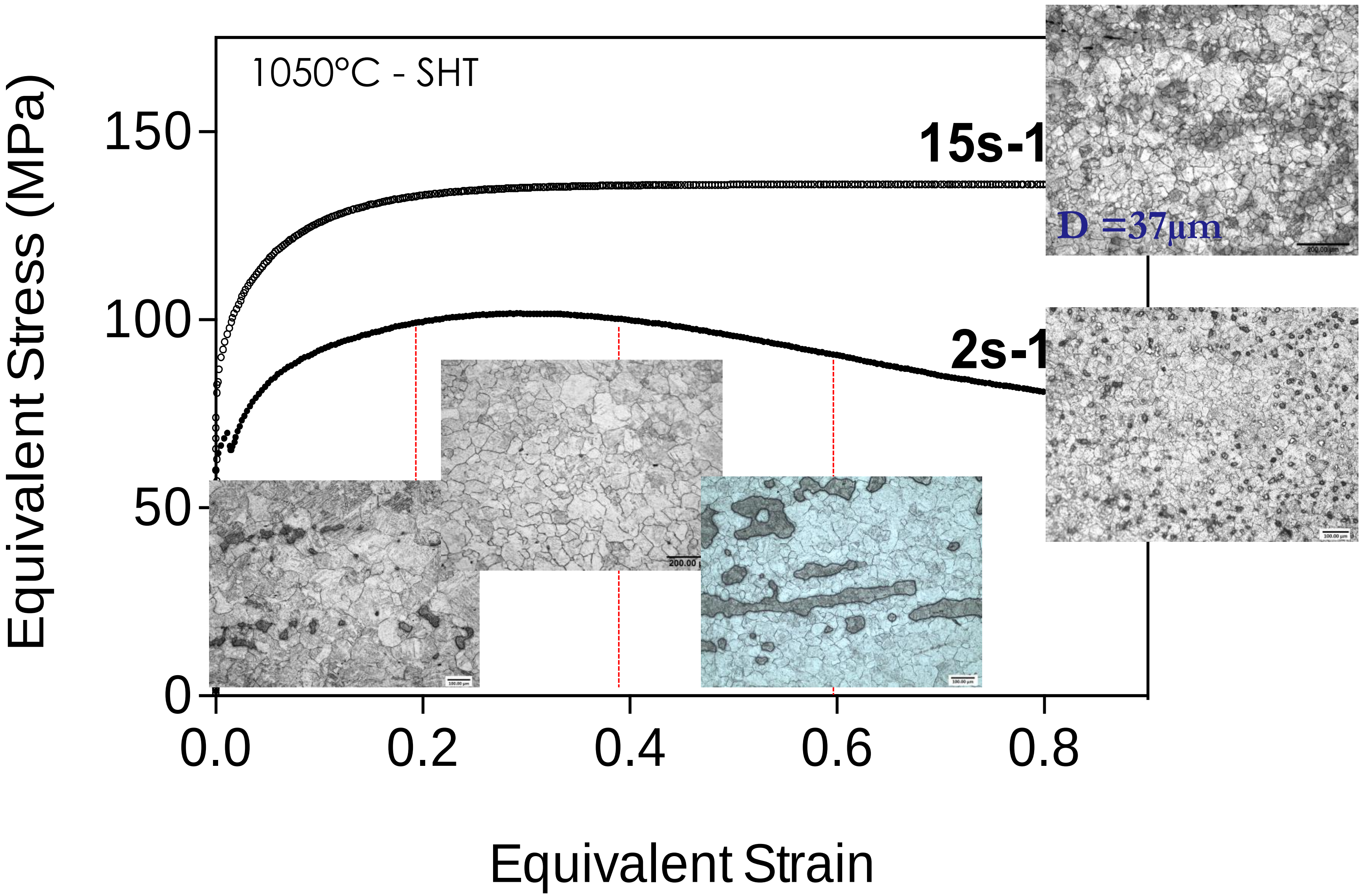




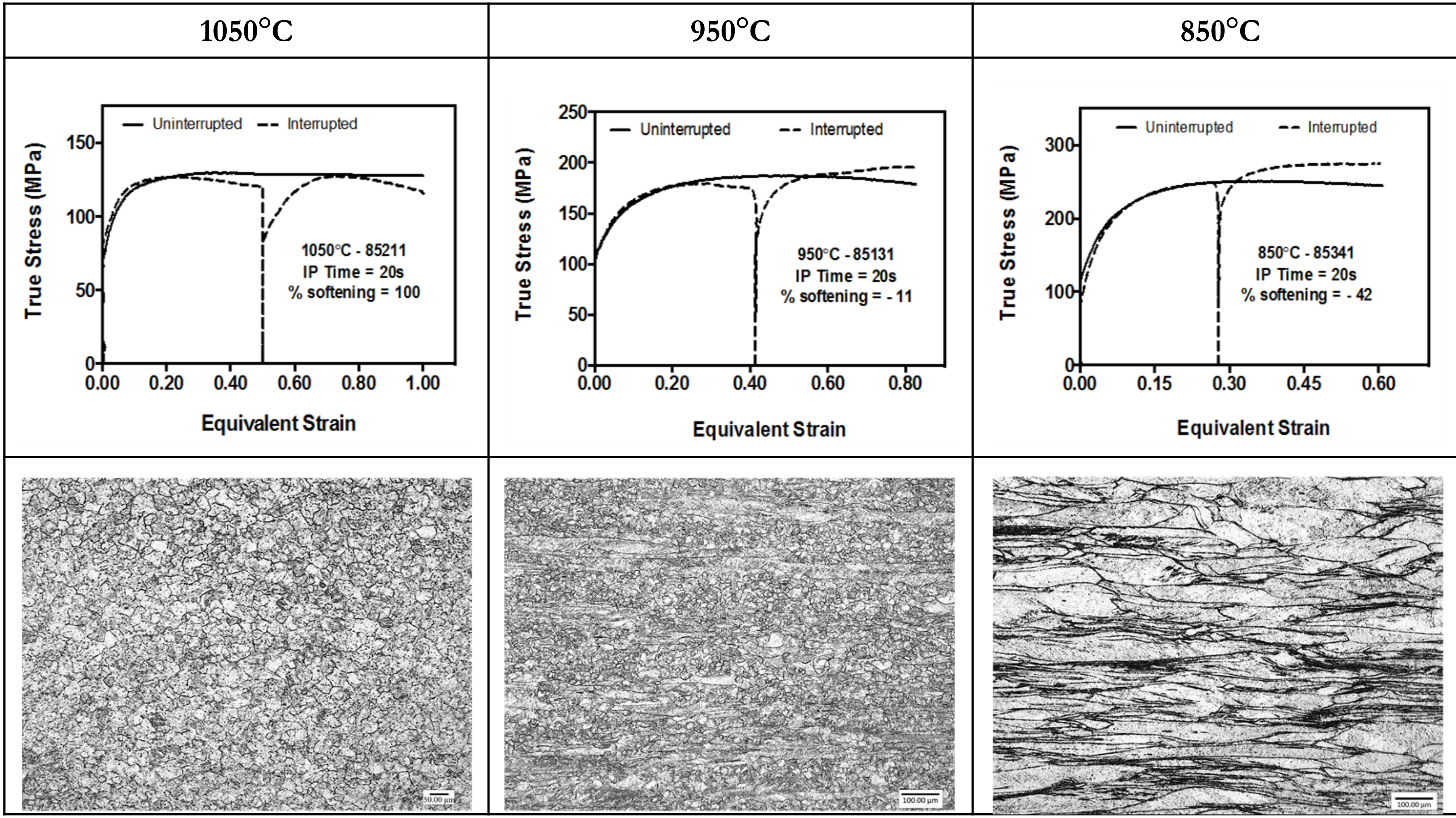
(d) Ɛ 0.40

Note : All tests were performed at constant stain rate of 2 s^{-1}

Effect of strain rate on DRX/DRV



C20Nb20 – Double Heat Tests (DHT)



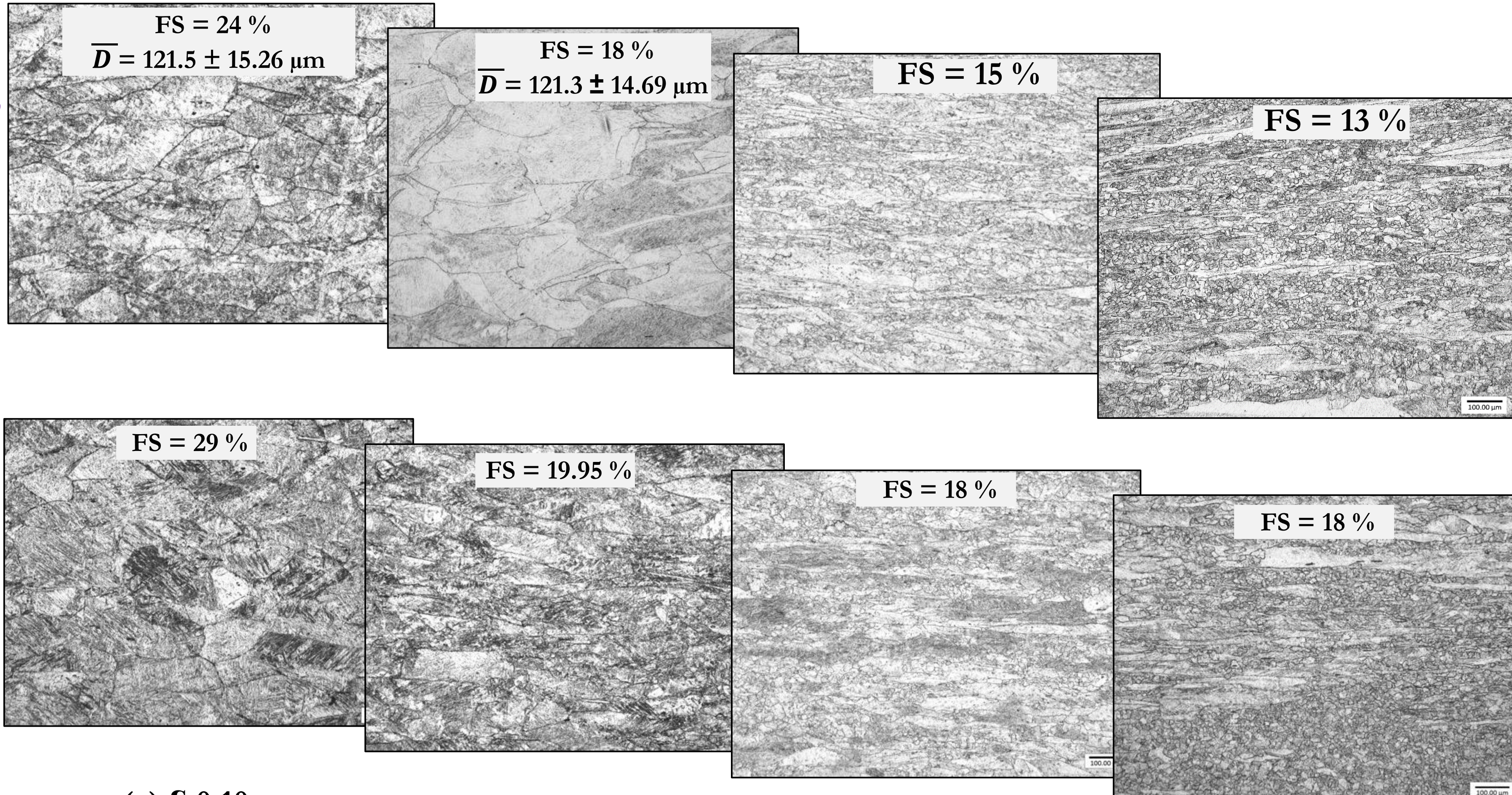
Equivalent stress-strain curves for DHT to a true strain (Ɛ) of 0.40

<u>Inter-pass time: 20s, Strain Rate: 15 s^{-1}</u>

950°C- IP 5s vs 20s

IP 5s

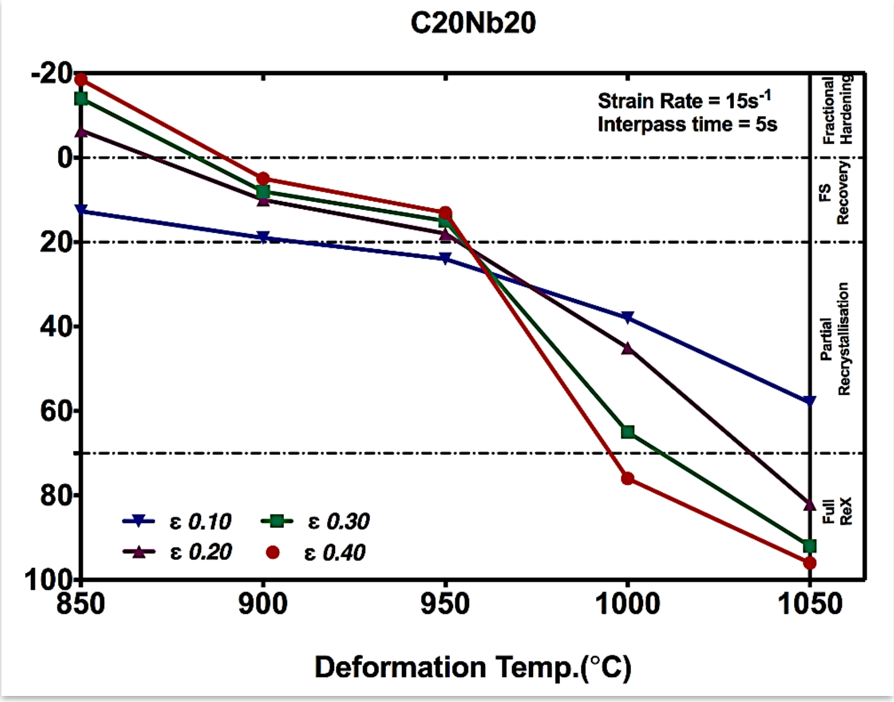
IP 20s



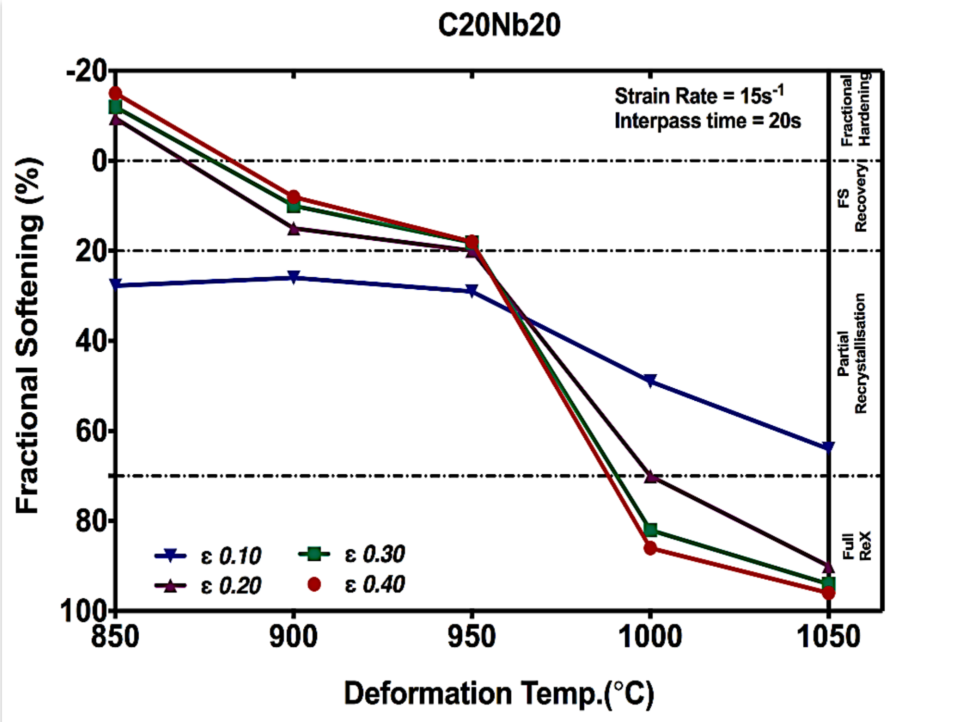
(a) ε 0.10 (b) ε 0.20 (c) ε 0.30 (d) ε 0.40

Note : All tests were performed at constant stain rate of 15 s^{-1}

Softening fraction as a function of deformation temperature

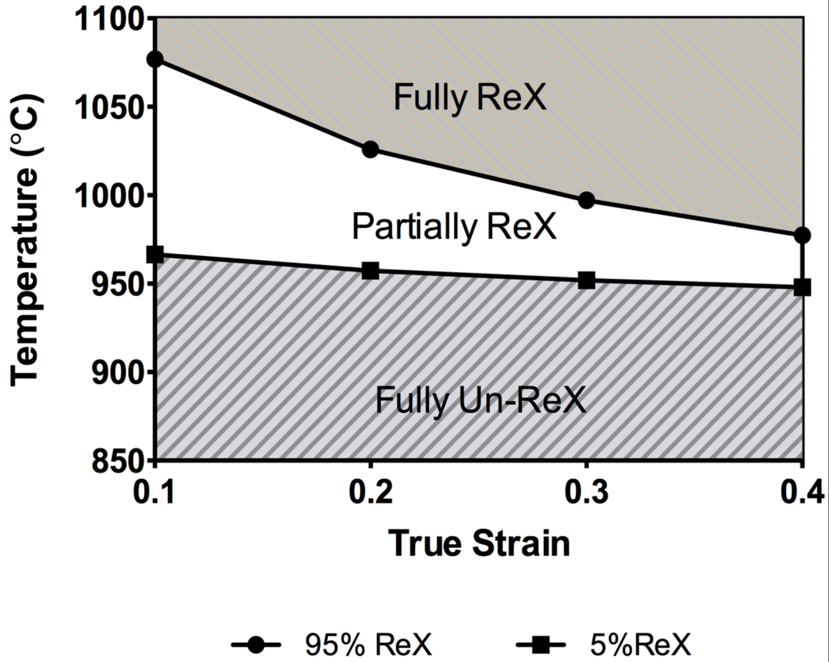
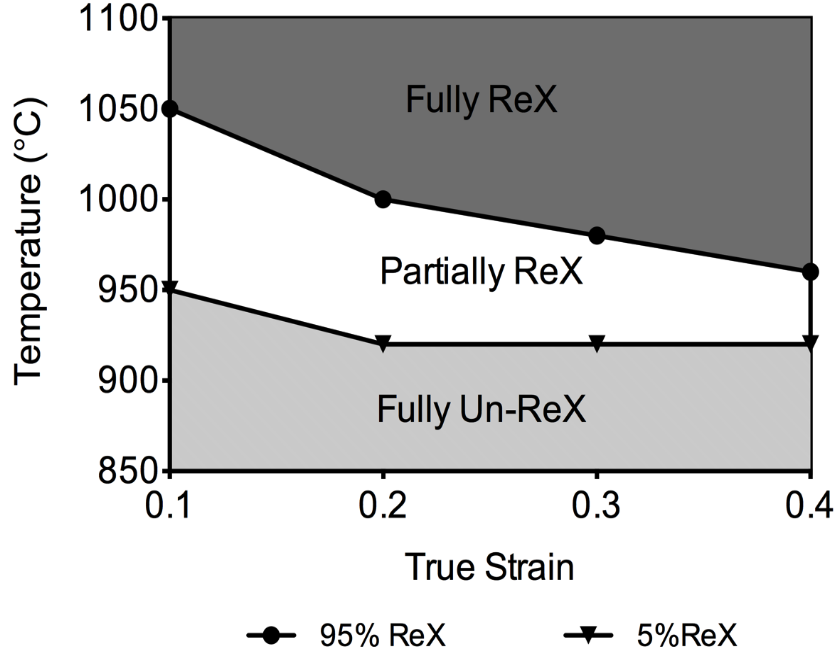


Inter-pass time: 5s



Inter-pass time: 20s

Recrystallisation Regime

Interpass time – 5s	Interpass time – 20s
	

<u>Softening Criteria</u>

$T_{5\%}$ ➔ 20 % softening ➔ due to recovery / Onset of Rex

$T_{95\%}$ ➔ 60 % softening + Metallography Observation

ReX and Precipitation Interaction

1. Time for 5 % recrystallisation, $t_{0.05X}$

$$t_{0.05x} = 6.75 \times 10^{-20}\, d_{o}^{2}\, \epsilon^{-4}\, exp\left(\frac{300000}{RT}\right)\, exp\left[\left(\frac{275000}{T} - 185\right)[Nb]\right]$$

2. Time for 5 % precipitation, $t_{0.05P}$

$$t_{0.05p} = A[Nb]^{-1}\varepsilon^{-1}Z^{-0.5}\exp\left(\frac{270000}{RT}\right)\exp\left(\frac{B}{T^{3}(\ln k_{s})^{2}}\right)$$

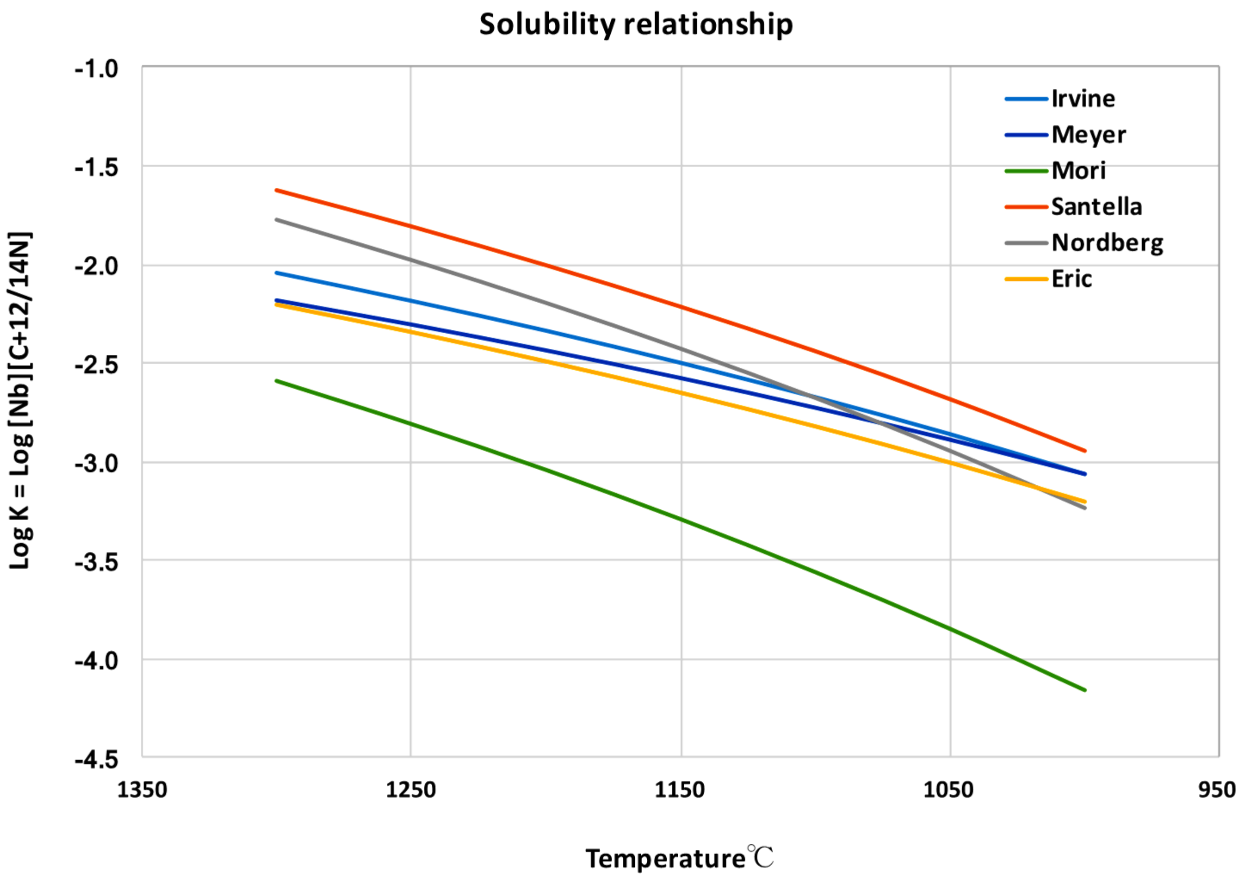
3. Recrystallisation fraction, JMAK Equation

$$X = 1 - \exp\left[\ln 0.95\ \left(\frac{t}{t_{0.05X}}\right)^{2}\right]$$

Ref. 1 C.M. Sellars, HSLA Steels 85, Beijing

Ref. 2 Dutta & Sellars, Materials Science and Technology, March 1987, Vol. 3

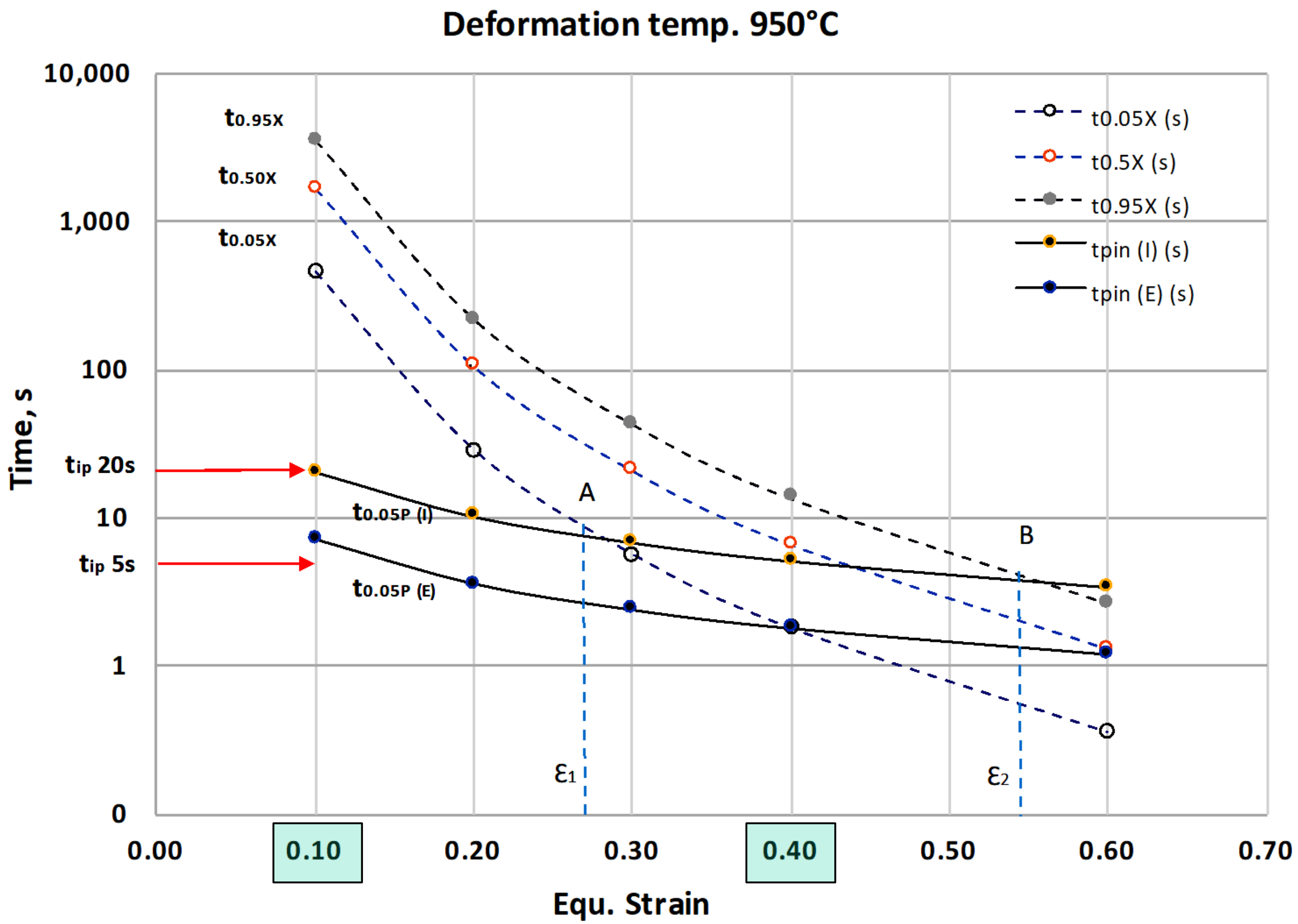
Solubility Equations



Irvine, 1967 $$\log[\mathrm{Nb}]\cdot[\mathrm{C}+12/14\mathrm{N}] = 2.26 - \frac{6770}{T}$$

Eric Palmiere, 1994 $$\mathrm{Log}\ [\mathrm{Nb}]\ [\mathrm{C}+12/14\mathrm{N}] = 2.06 - \frac{6700}{T}$$

Interaction bet^n REX & PPT KINETICS

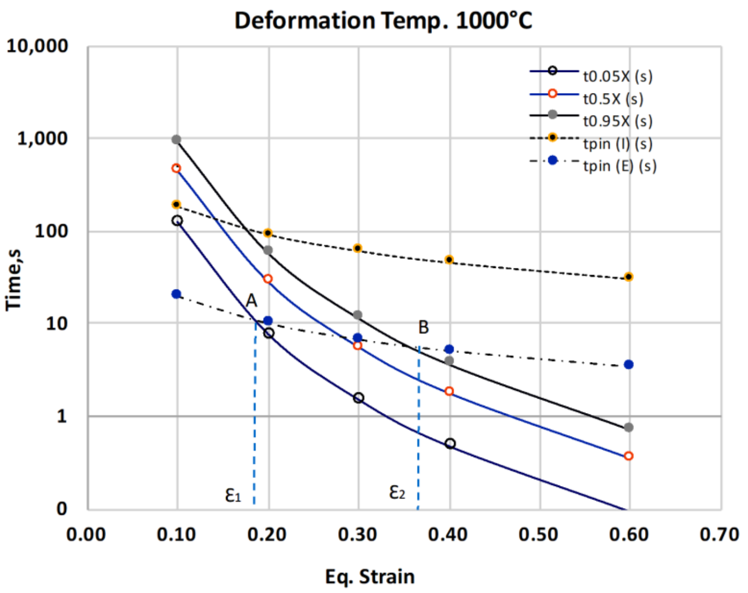


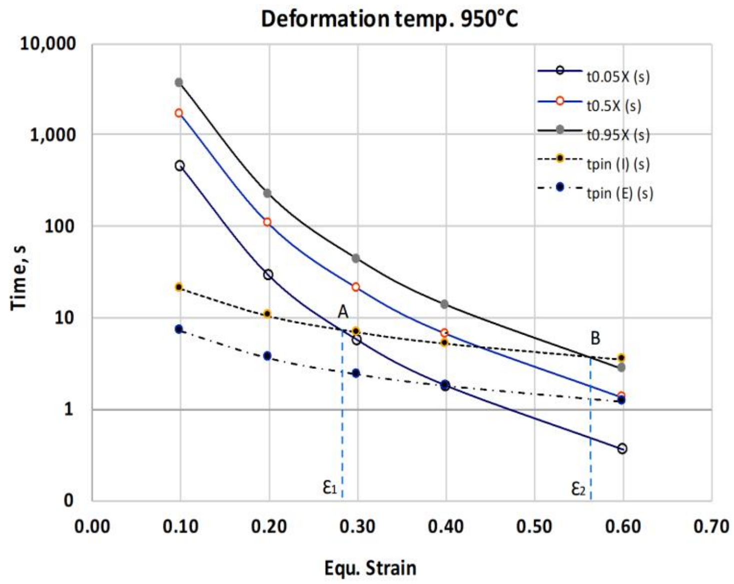
For Ɛ < Ɛ1, the PPT is initiated before REX. $t_{0.5X}$ and $t_{0.95X}$ are shifted to much longer times.

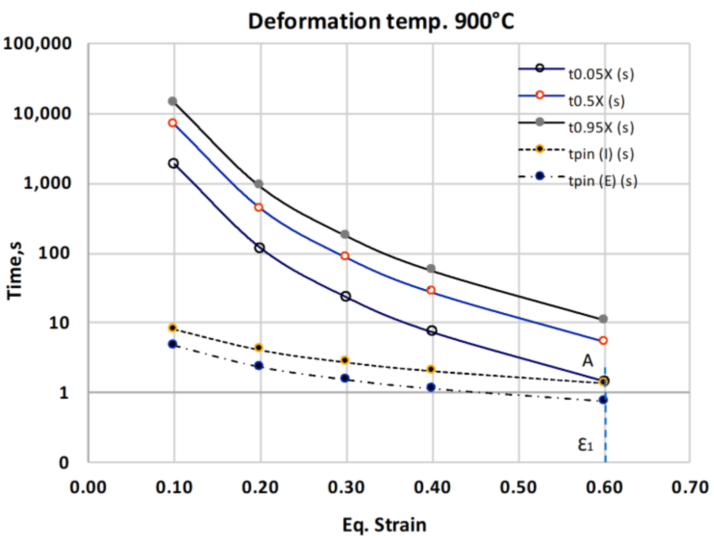
For Ɛ1 to Ɛ2 , the REX is initiated before PPT but its completion will be delayed by PPT.

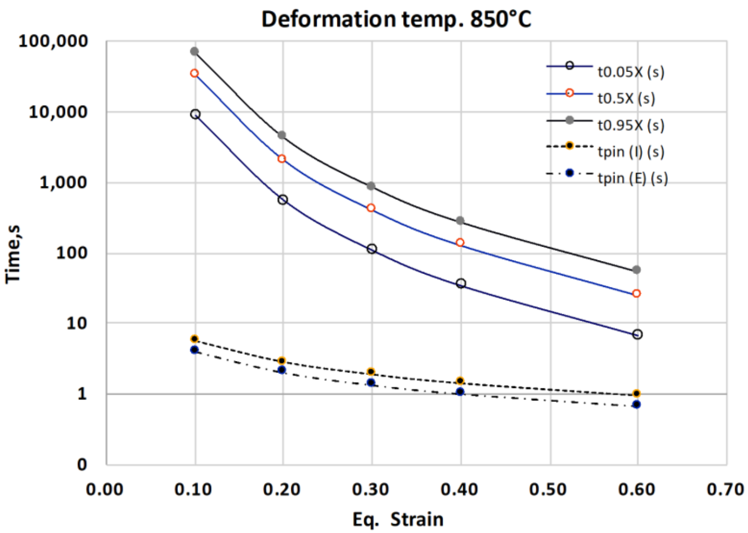
For Ɛ > Ɛ2 , the REX is completed before PPT can be initiated.

Interaction bet^n REX & PPT KINETICS

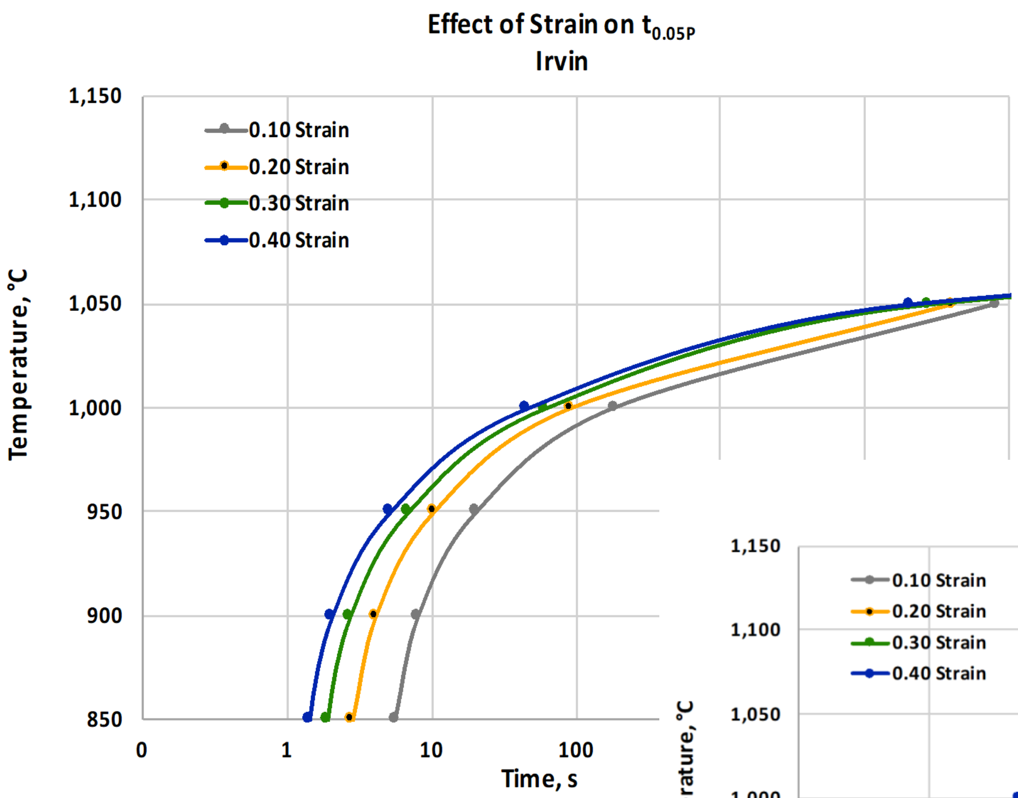


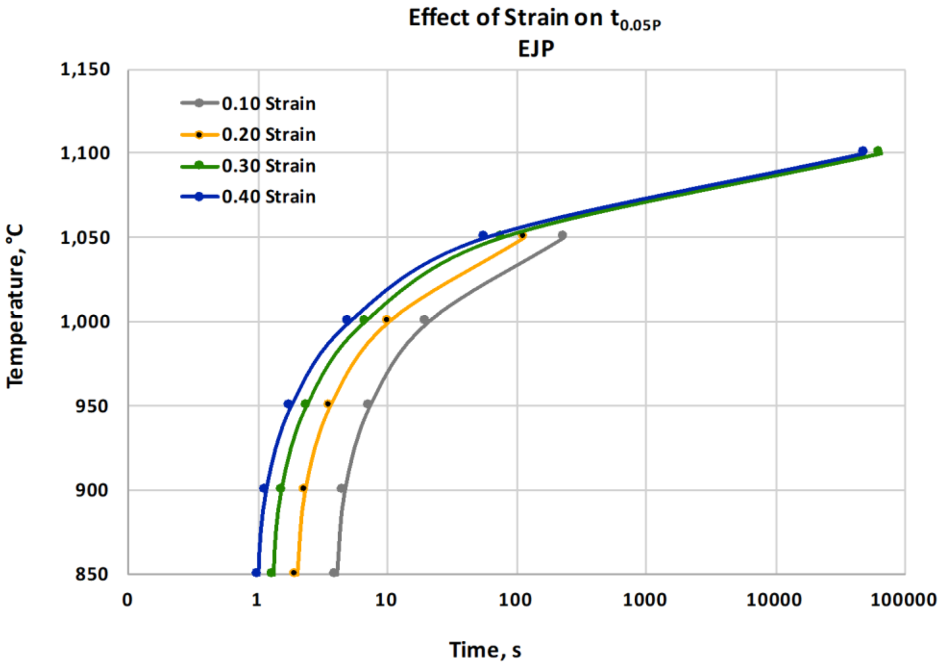




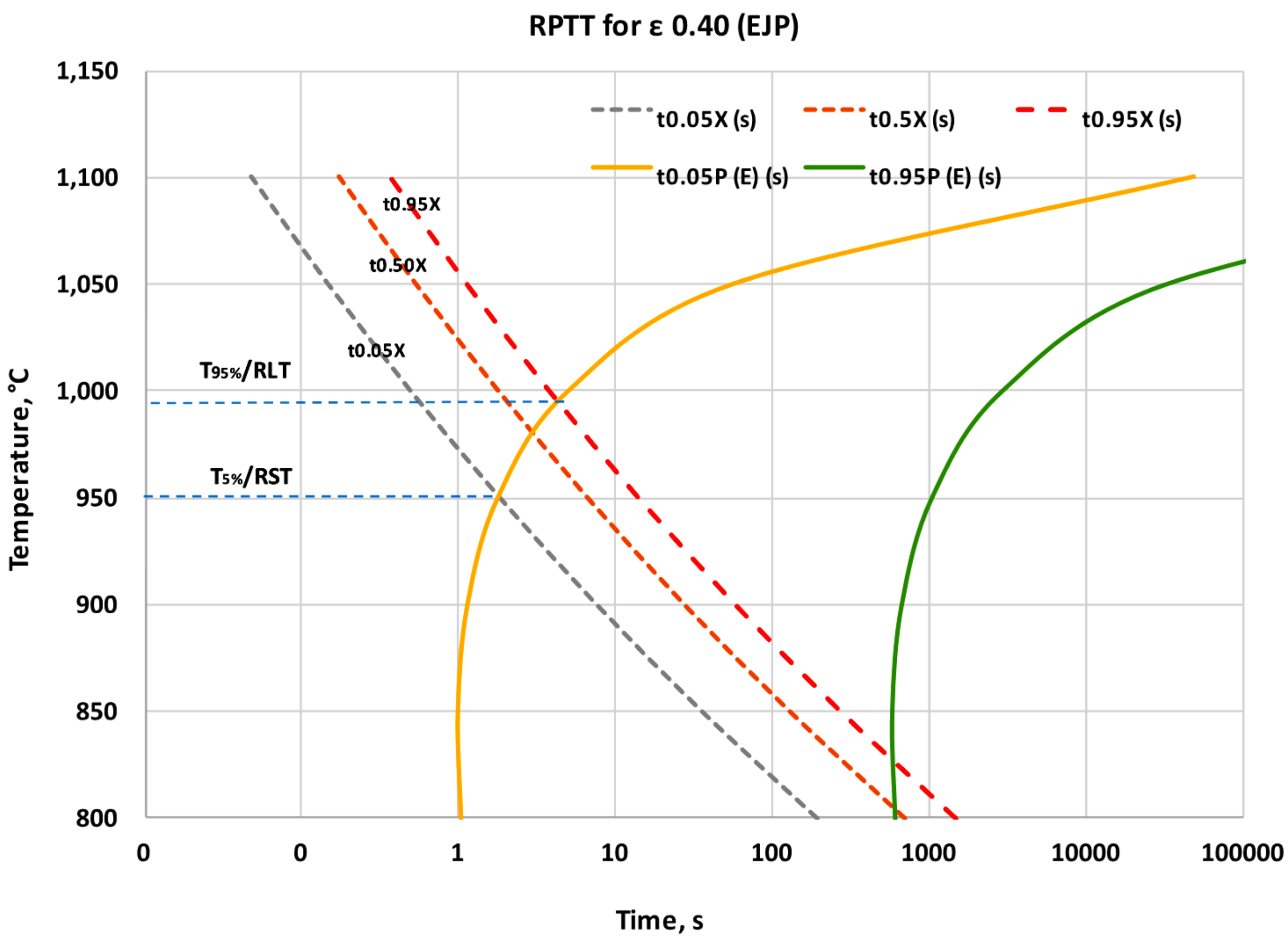


Effect of applied strain on $t_{0.05P}$





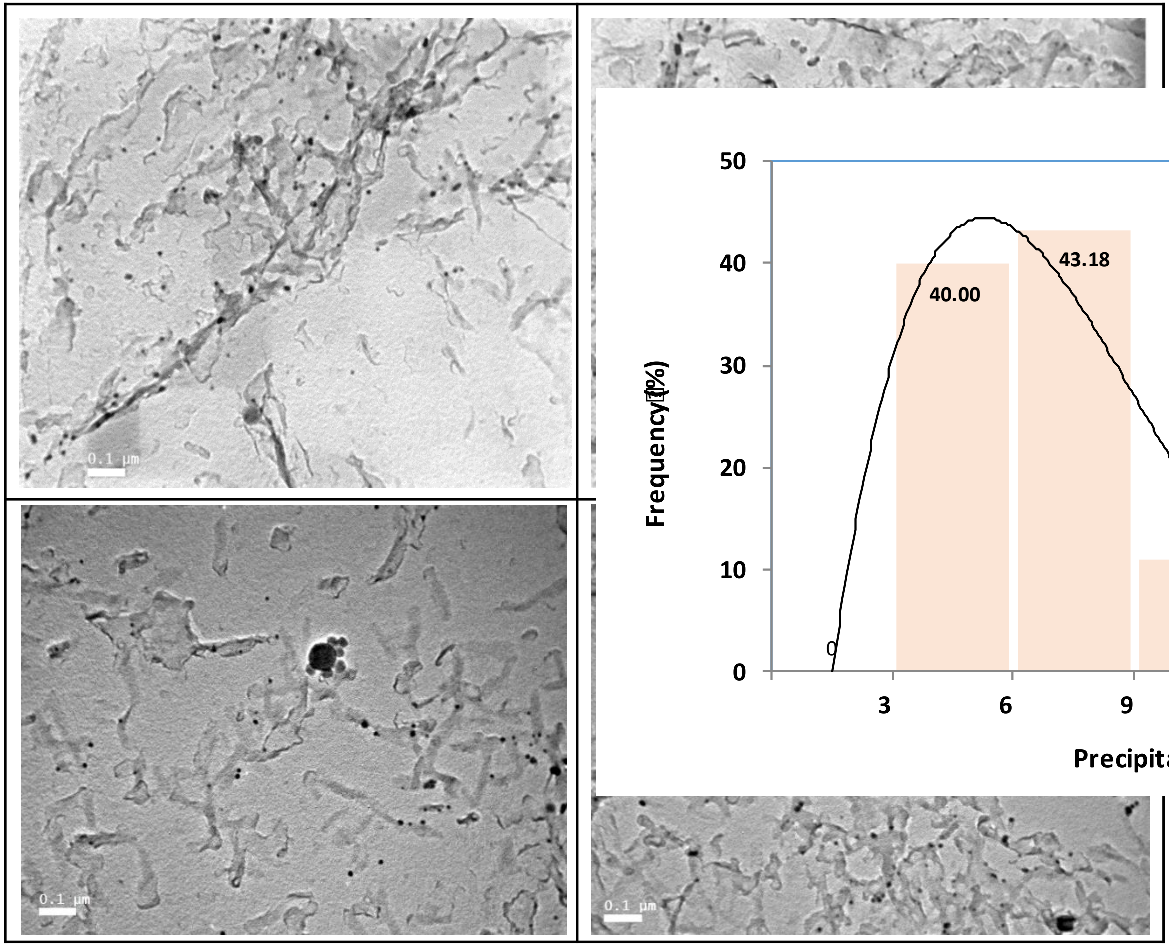
RPTT Analysis

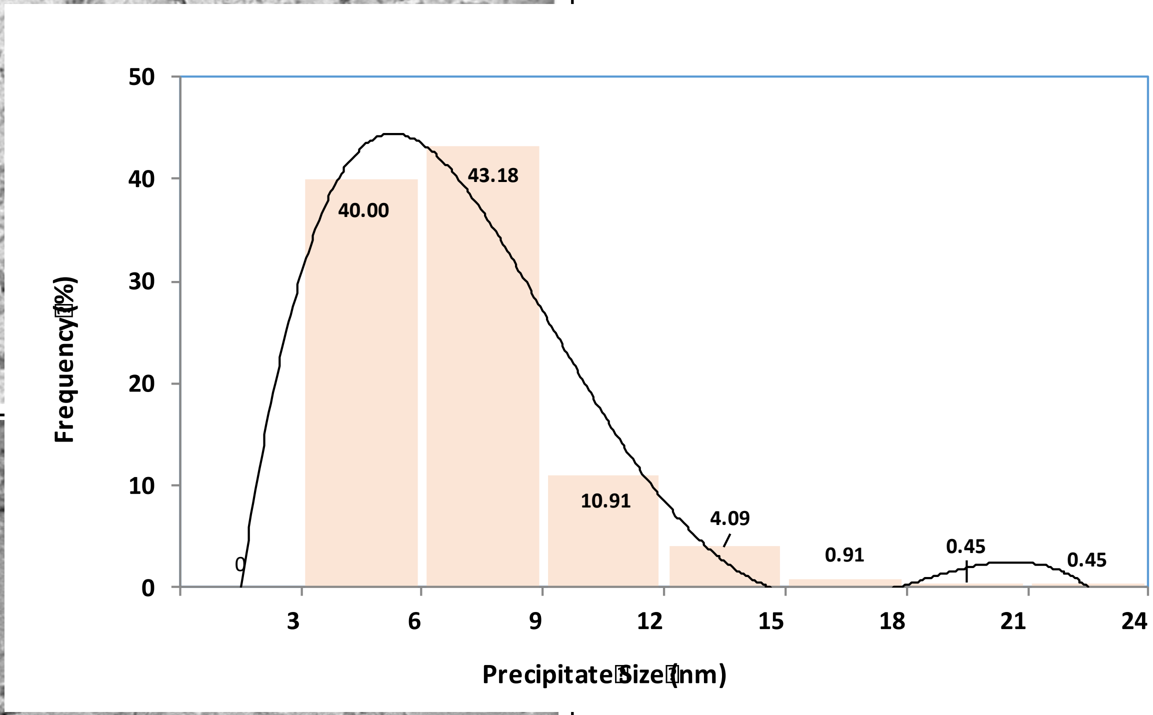


Precipitation start and finish times based on EJP solubility equation.

TEM Analysis – Extraction Replica

C20Nb20 - 950°C – ε 0.40, IP time: 20s





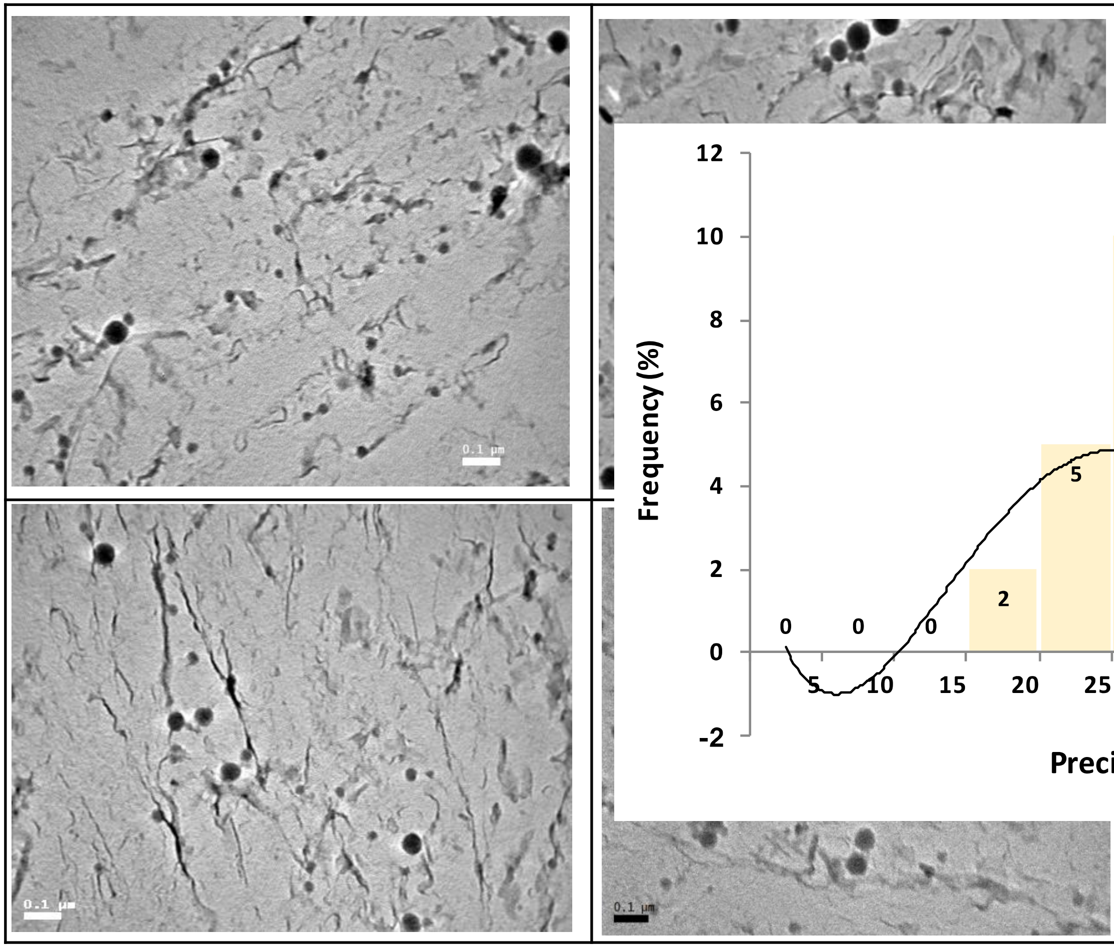
Typical Size Distribution

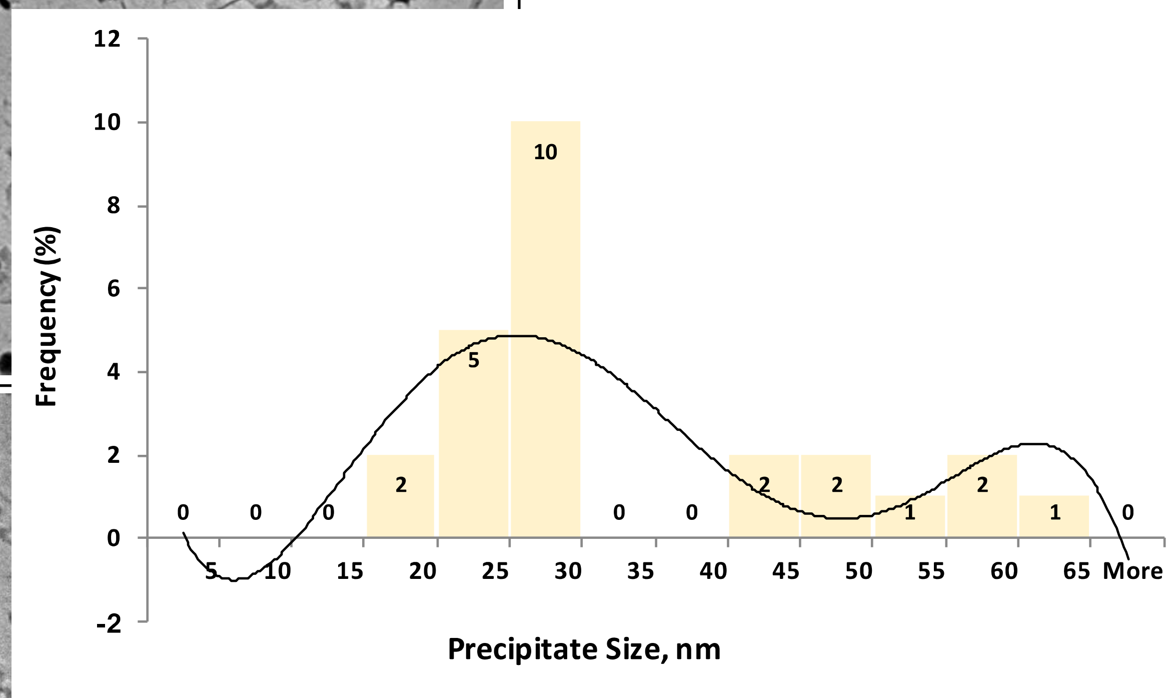
D_m= 8.38 ± 0.41 nm

(measurement of 1537 particles over area of 8.18 µm^2)

TEM Analysis – Extraction Replica

C20Nb20 - 950°C – ε 0.10, IP time:20s





Typical Size Distribution

D_m= 25.8 ± 0.85 nm

(measurement of 140 particles over area of 3.8 μm^2)

Ongoing work

1. TEM studies on 0.20 % C steels to determine precipitation kinetics and volume fractions so that comparison between F_{RXN} & F_{PIN} can be done.

2. Recrystallisation studies on 0.80 % C steels with similar levels of Niobium and experimental matrix. (Master student : Recrystallisation studies on 0.40 and 0.60 % C steels for interpass time of 20s).

3. We will have softening data for carbon ranging from 0.20 to 0.80 % C and effect of Nb from 50 – 200 ppm. It could be one of the most comprehensive studies on recrystallisation and precipitation interaction in high carbon steels.

4. Take forward the earlier modelling work at Sheffield and applied to current experimental steels

Conclusions

1. The fraction softening increases with increase in deformation temperature for a constant strain rate and interpass time conditions.

2. An increase in strain leads to increase in fraction softening for any given deformation temperature.

3. The fraction hardening observed for deformation temp. < 950°C is attributed to the strain induced precipitation of Nb(C,N) in deformed austenite and subsequent inhabitation of recrystallisation.

4. The $T_{5\%}$ and $T_{95\%}$ (REX window)temperatures determined by applying the softening criteria and quantitative microscopy goes well with industrial observation.

5. The dilute Addition of 170 ppm of Nb has significant impact on static recrystallisation behaviour !

Thank you..

Q & A