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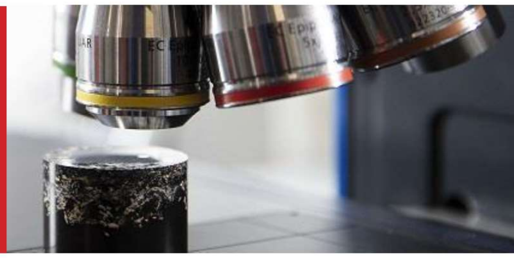
TESTING
MATERIALS & ENGINEERING



Microstructural Parameters and Creep Exposure for 9Cr Steels. A Tentative Quantitative Correlation Based on "Metallic" Replication

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Background

- It is now widely accepted that cavitation is not a safe criterion to assess creep damage in 9Cr martensitic steels
- In Italy, the first power plants with Grade 91 components are about to reach the first 100000 operation hours
- It is necessary to define an alternative or complementary experimental methods to classic morphological/cavitational replicas in order to assess creep damage
- Submicrostruttural analysis on metallic components
  - Extraction replica (TEM)
  - Metallic replica (SEM)

Approaches Proposed for In Service Inspection

For creep damage assessment: Microstructure documentation based on 2 potentially applicable Approaches:

- Method 1: analysis of particle size, number
- Method 2: analysis of subgrain size

Grade P91: Look For Particles

Particle type	Typical metals	Typical Non-metals	Possible compounds
Primary MX	Nb, V…. Mo, Ti	C, N,…. B	Ti(C,N), Nb(C,N)
Secondary MX	Nb, V…. Mo, Ti	C, N,…. B	NbN, NbC, VN, VC, MoC V(C,N), Nb(C,N)
$M_{23}C_6$	Cr, Mo, V, Fe, Nb	C	$(Cr_{16}Mo_3V_1(Fe,Nb)_3)C_6$ $Cr_{23}C_6$
M_6C	Mo, V, Nb, Cr, Fe	C	$(Mo_4CrV)C$
M_2X	Cr, V, Fe	C, N	
Laves	Fe, Cr, Mo, W, Si	-	$(FeCr)_2(WMo)$
Z	Cr, V, Nb	N	Cr(V,Nb)N

Strengthening particles

Strength reducing particles

Grade P91: Particles

MX / $M_{23}C_6$ → strengthening effect:

- Interact with dislocations, increasing the load required to:
  - a) move the dislocations
  - b) generate further dislocations
  - c) overcome the particles

- Stabilize the surrounding (martensitic) microstructure :
  - a) avoid grain boundary migration/sliding
  - b) reinforce or attract substructures

MX/$M_{23}C_6$: Guarantee material creep strength

Grade P91: High Temperature Exposure ONLY

Effects of hot exposure ONLY → diffusion:

- MX:
  - Decrease in number and increase in size;
  - Lose coherence to the matrix;
  - transform into Z (depending on finite T and d);
- $M_{23}C_6$:
  - Decrease in number and increase in size
  - migrate to grain boundaries;
- Laves
  - Will nucleate with time
  - Bind hardening elements (impoverish the matrix)
  - Decrease in number and increase in size
- Martensite sub structures (typically acicular) will tend to enlarge and round.

To all of this we have to add…

Grade P91: High Temperature Exposure WITH LOAD

…mechanical load: ➔

- elastic strain: lattice slightly increases in size;
- plastic strain: generation and movement of dislocations and generation of new substructures;
- direct and accelerate to diffusion (increased core-diffusion contribute).

Mechanical load makes ageing processes faster

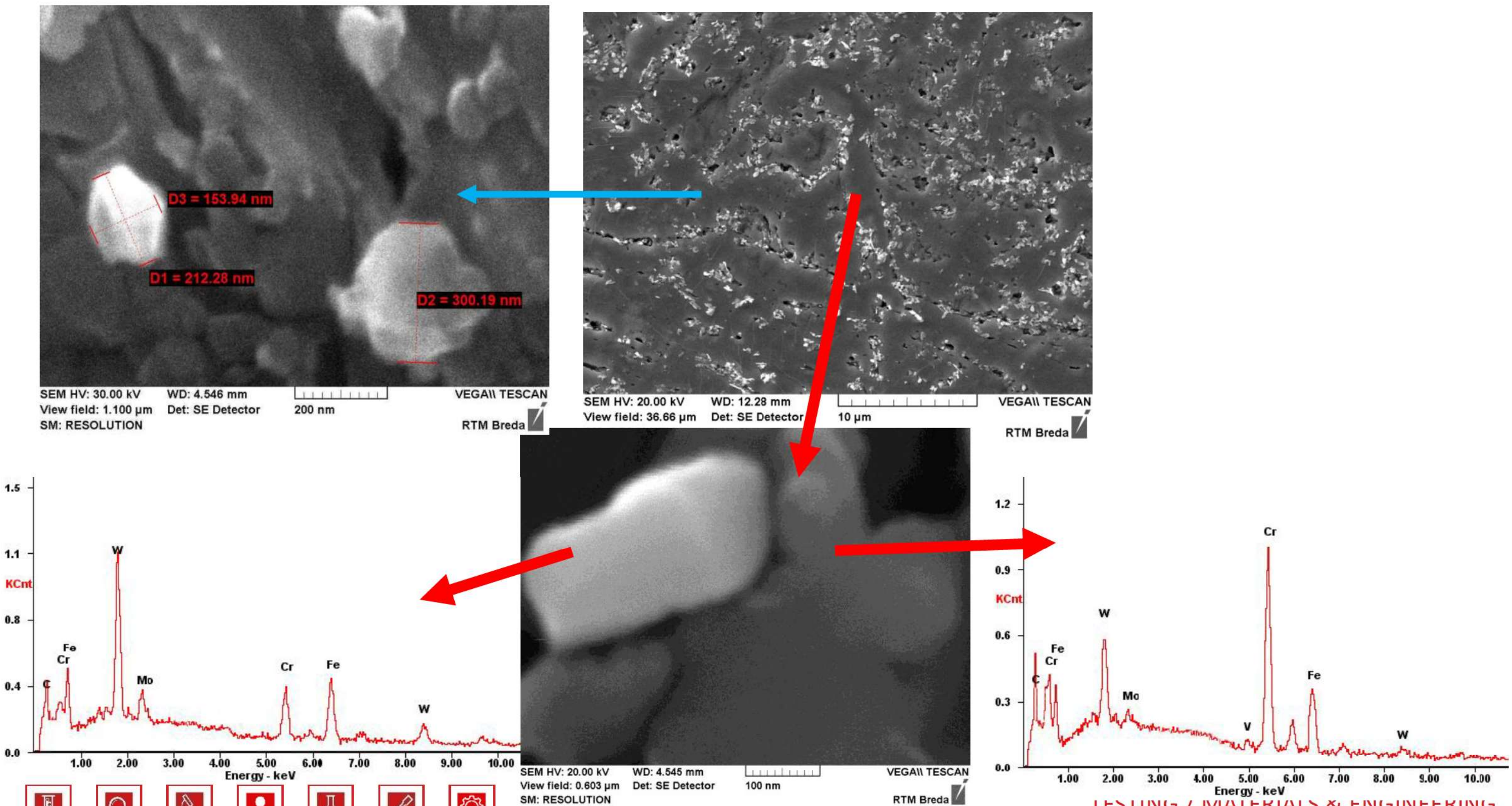
Due to the strong interaction between dislocations and tiny particles, «creep damage» will be mainly correlated with MX particle evolution

Method 1
Particle Analysis

- Study of the interaction between particles and dislocations to estimate rupture at (constant) service temperature
  - Starting conditions: particle sizes and distribution;
  - Intermediate on site microstructure assessments: particle sizes at known temperature, load, time;
  - End-of-Life criterion: limit line beyond which rupture has to be expected.

Examples of Metallic Replicas

- Es: measure of particle size and identification of chemical composition on P92 steel



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Equations

$$\dot{\varepsilon}_{min} = \frac{A(T,\sigma)}{t_u}$$

Monkman Grant

$$\dot{\varepsilon}_{min} = C \left(\frac{\sigma - \sigma_i}{G(T)}\right)^n exp\left[\frac{-Q}{RT}\right]$$

Norton-Dorn

$$\sigma_i = \alpha \frac{G(T)\, b}{l_0} ln\left[\frac{D}{2b}\right]$$

Orowan

$$D = \sqrt[3]{K_d(T,\sigma)\,(t - t_0) + D(t = t_0)^3}$$

Ostwald Particle growth

Valutazione del danno e della vita residua

We assume that:

- For constant stress and temperature, particle diameter increases only as a function of time under load and initial diameter

- The influence of a particle on strength at a given load is proportional to particle size and temperature ➔ we can define a «temperature corrected diameter»

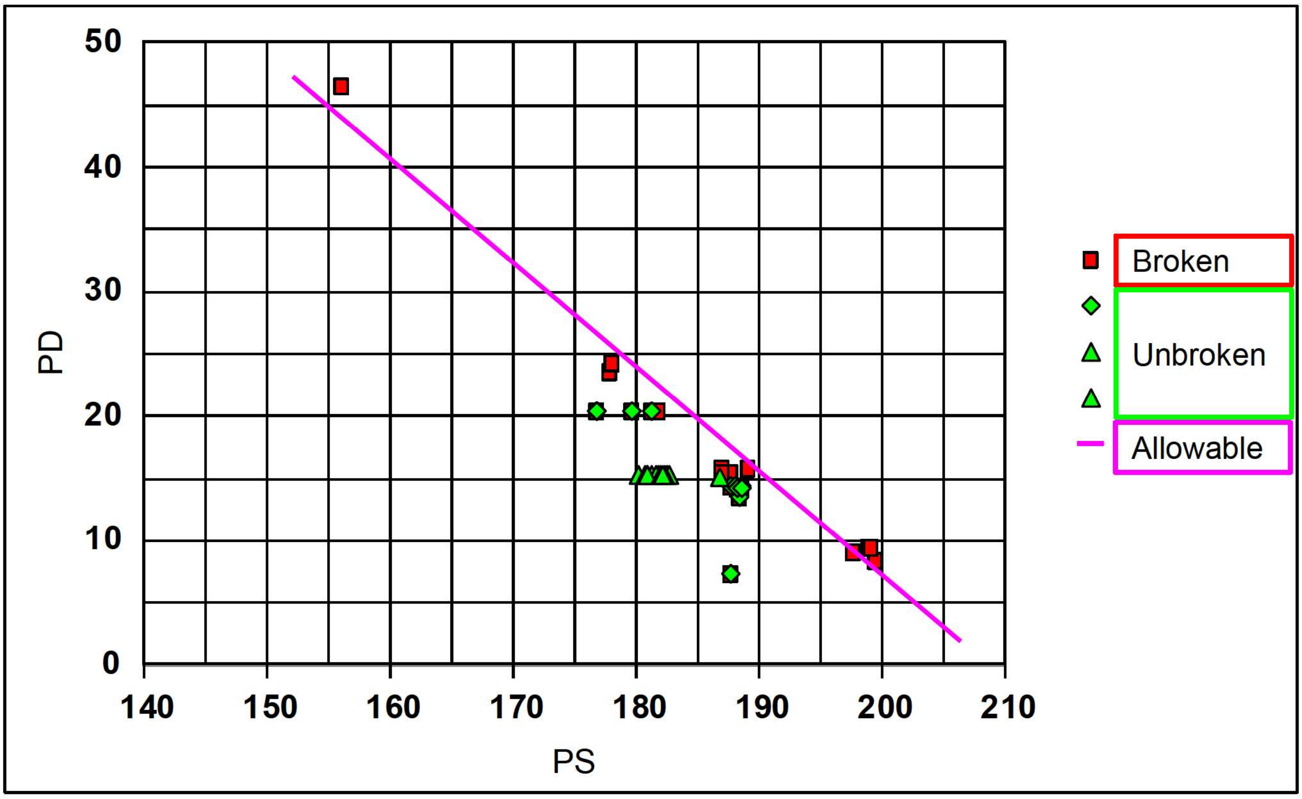
$$PD = (T)\ (20 + log[D])/100$$

- A change in diameter leads to a change in creep rate (so an «acceleration»). Average acceleration is estimable through minimum creep rate and current time, so that we can derive the parameter PS

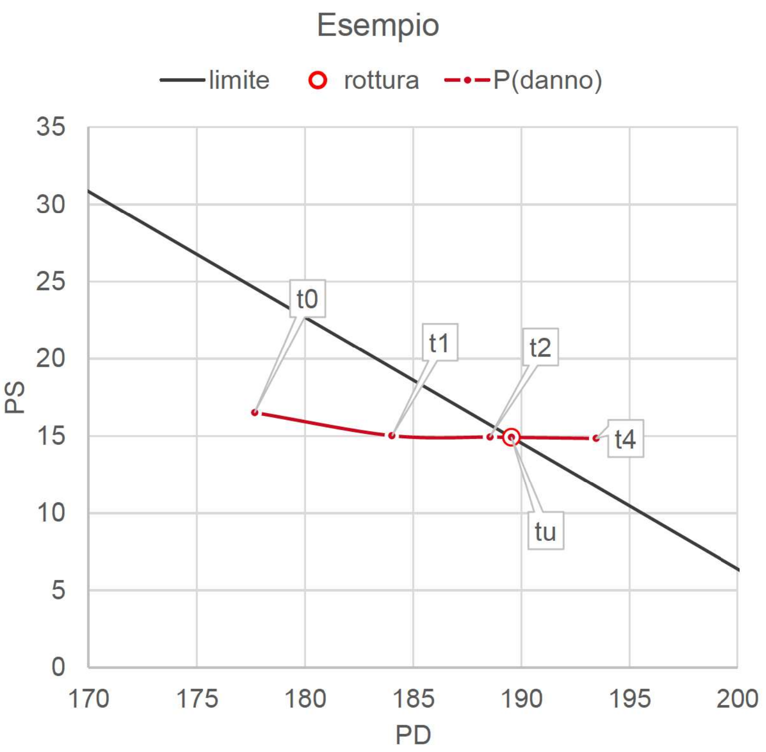
$$\ddot{\epsilon}_{min} = \frac{A\sigma^n}{t}$$

$$PS = log[\ddot{\varepsilon}] = A(n\ log[\sigma] - log[t])$$

PS-PD diagram



Example



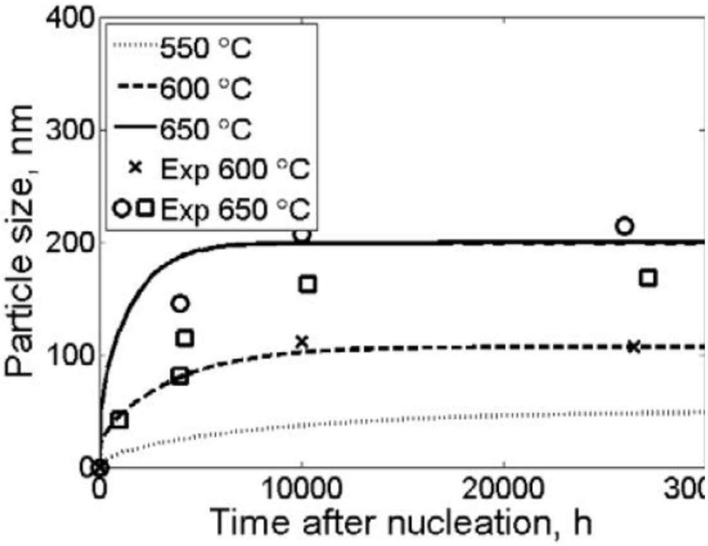
- t_0: initial state
- $t_1 \rightarrow t_4$: diameter growth (assumed parabolic)
- t_u: time at limit line crossing

Method 2: Substructure analysis

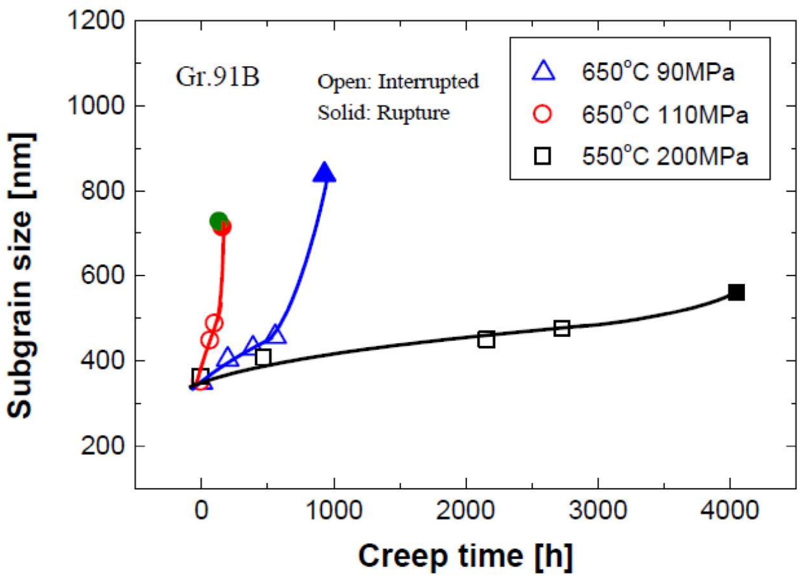
- Technical reasons
- Analysis based on literature data and internal tests

Technical reasons

- Is particle evolution and growth always significant for creep damage? Asymptotic growth is observed, in some cases.
- A typical witness of ongoimg creep damage is subgrain size, apparently always increasing.

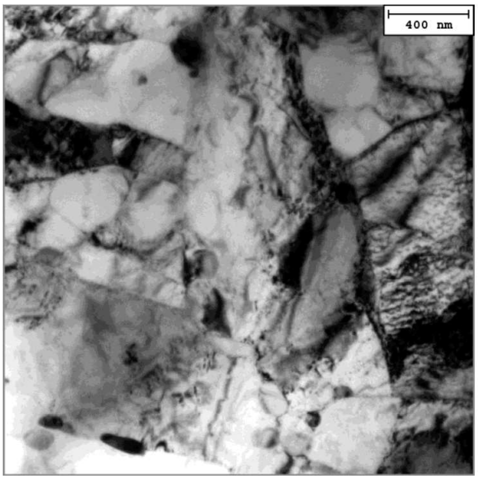


H. Magnusson, *Creep modelling of particle strengthened steels,* Stockholm, 2007

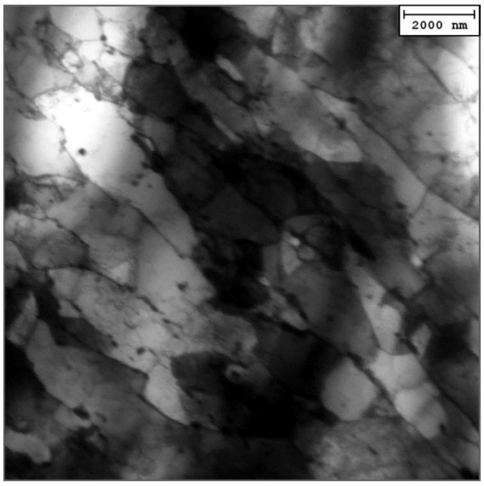


R. Chen, *Microstructural Degradation during High Temperature creep of Mod.9Cr-1Mo Steel and Its Application to Life Assessment,* Tohoku University, 2011.

Subgrain size



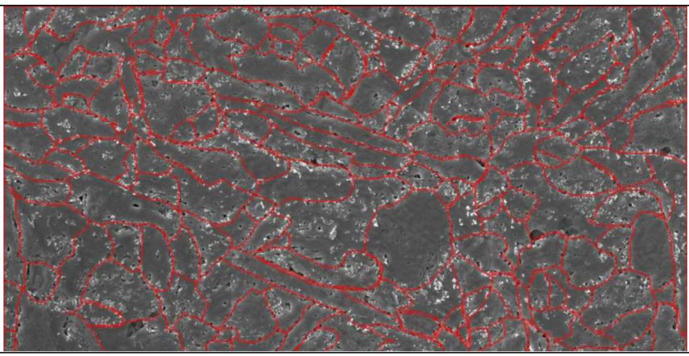
At test start



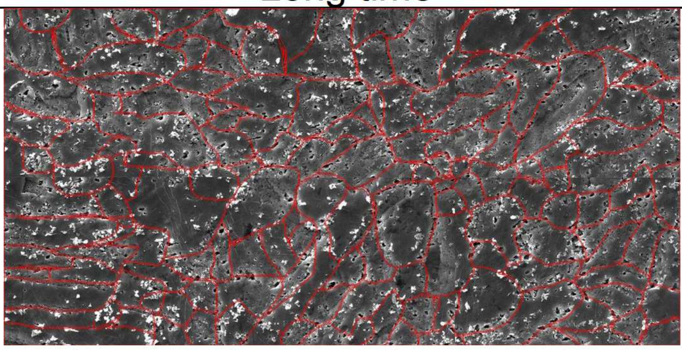
After 600°C creep test

Metallic replica

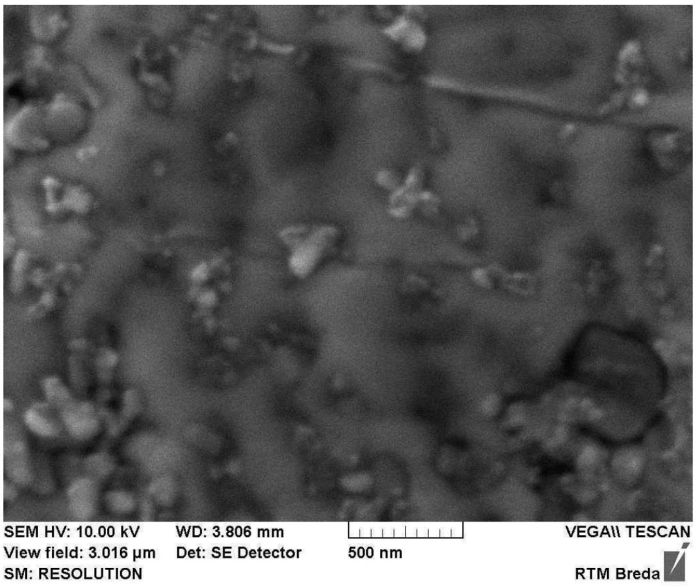
Short time



Long time



statistica di tutti i domini microstrutturali	DIAMETRO MEDIO		FERET MEDIO		ASPECT		LATO EQUIVALENTE	
	tempo corto	tempo lungo	tempo corto	tempo lungo	tempo corto	tempo lungo	tempo corto	tempo lungo
	µm		µm		≥1		µm	
Min	1,7	1,8	2,0	2,0	1,0	1,0	1,2	1,6
Max	14,0	17,9	63,2	22,3	15,4	8,6	5,3	11,5
Mean	5,6	6,3	7,4	8,0	2,4	2,3	2,0	5,6



Equations

- Damage parameter, derived from Norton-Dorn law

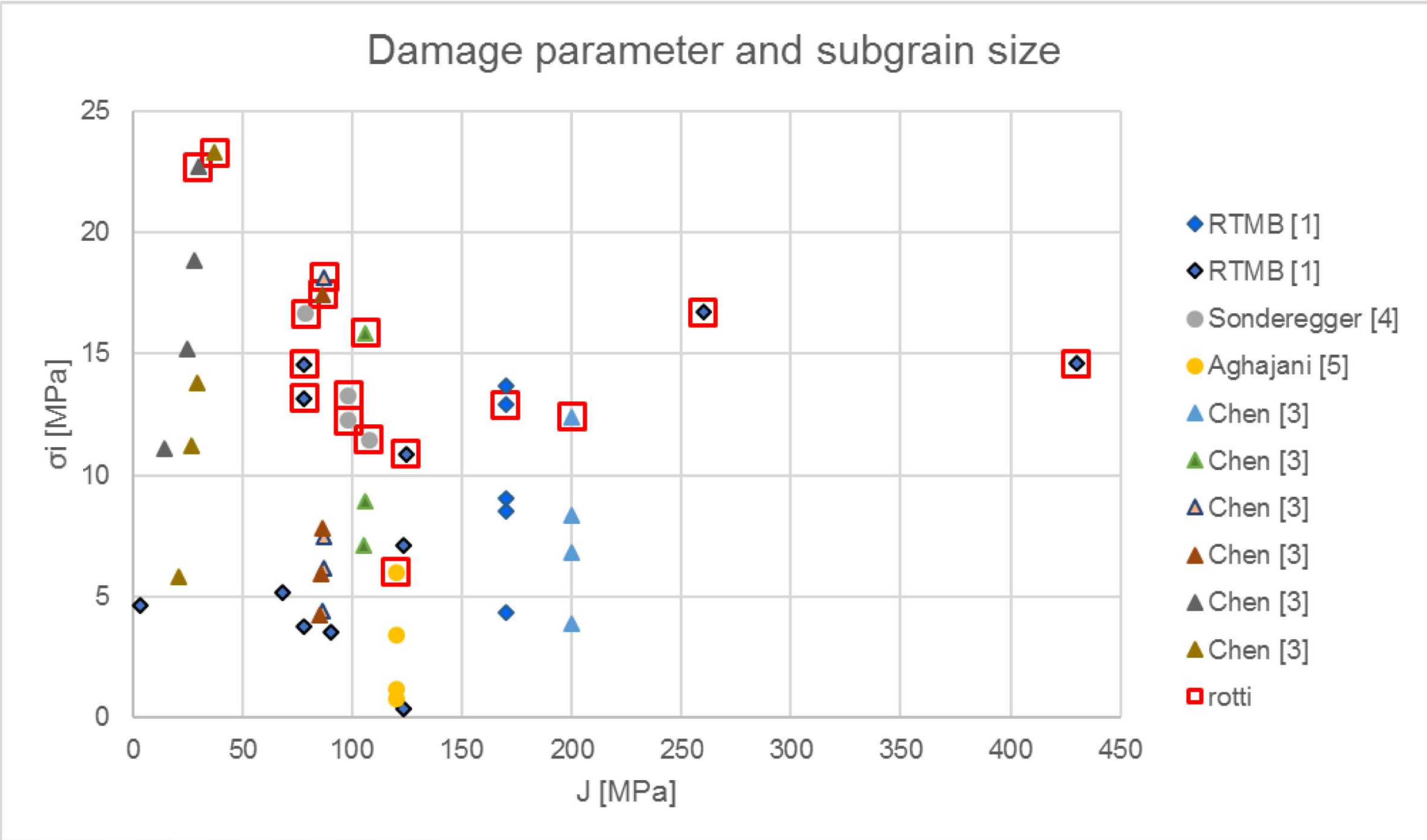
$$\dot{\varepsilon}_{min} = C \left(\frac{\sigma - \sigma_i}{G(T)}\right)^n exp\left[\frac{-Q}{RT}\right]$$

$$J = \sigma - \left(\frac{C^*}{t} \cdot \frac{1}{exp\left(\frac{-Q}{kT}\right)}\right)^{\frac{1}{n}}$$

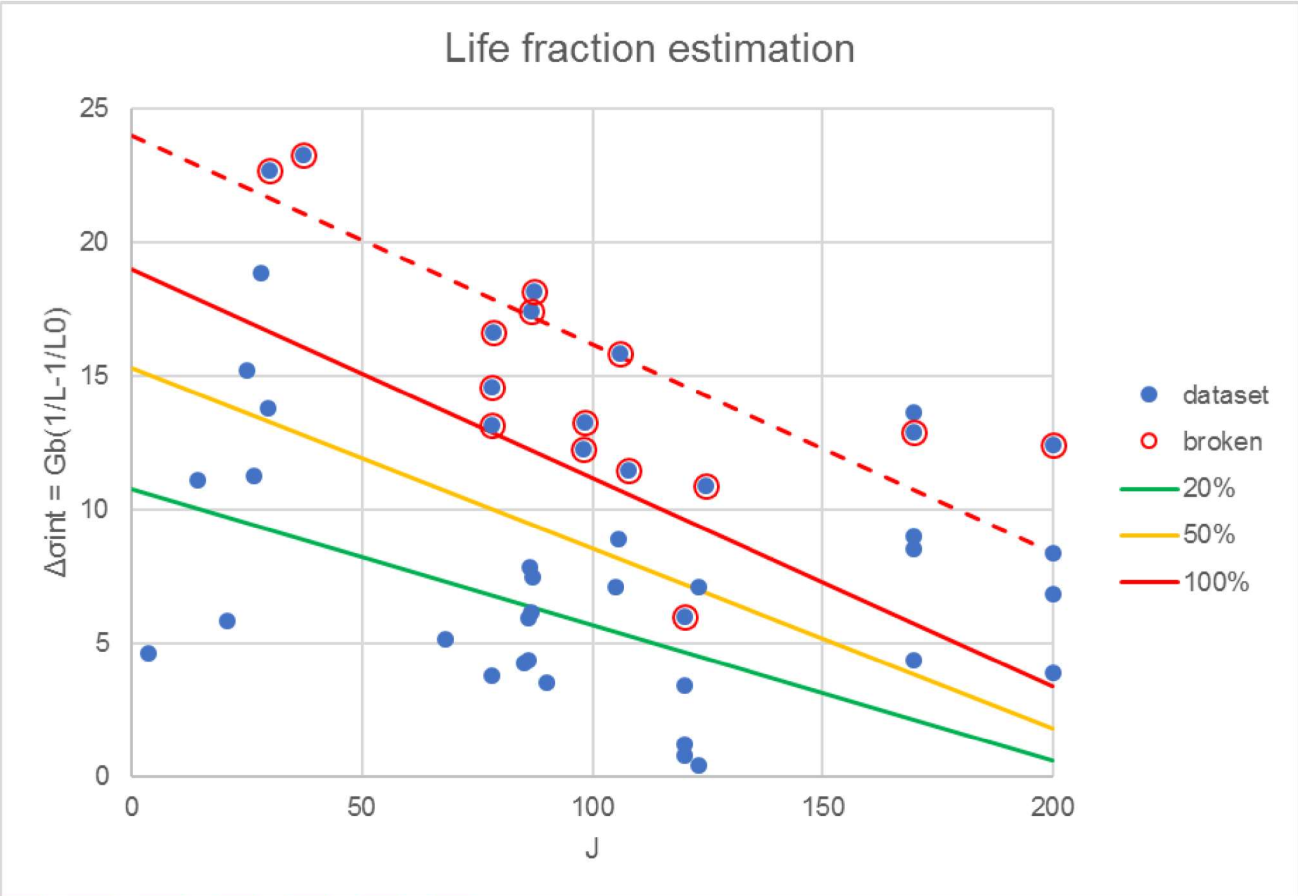
- Internal stress

$$\sigma_i = \alpha\, G(T)\, b \left[\frac{1}{\lambda_0} - \frac{1}{\lambda}\right]$$

Analysis of interrupted creep tests



Damage assessment and expended life fraction



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Conclusions

- Two proposals for an experimental method to estimate creep damage of operated 9Cr martensitic steel componets
  - Method 1 based on particle evolution
  - Method 2 based on sub-microstructure evolution
- Both work with in-field metallurgical investigation methods
  - Metallic sample taken off the comoponent ➔ TEM/SEM
  - Convenbtional resin foil baed extraction replicas ➔ TEM
  - Metallic replicas ➔ SEM
- Strong need of data to strengthen method and correlation

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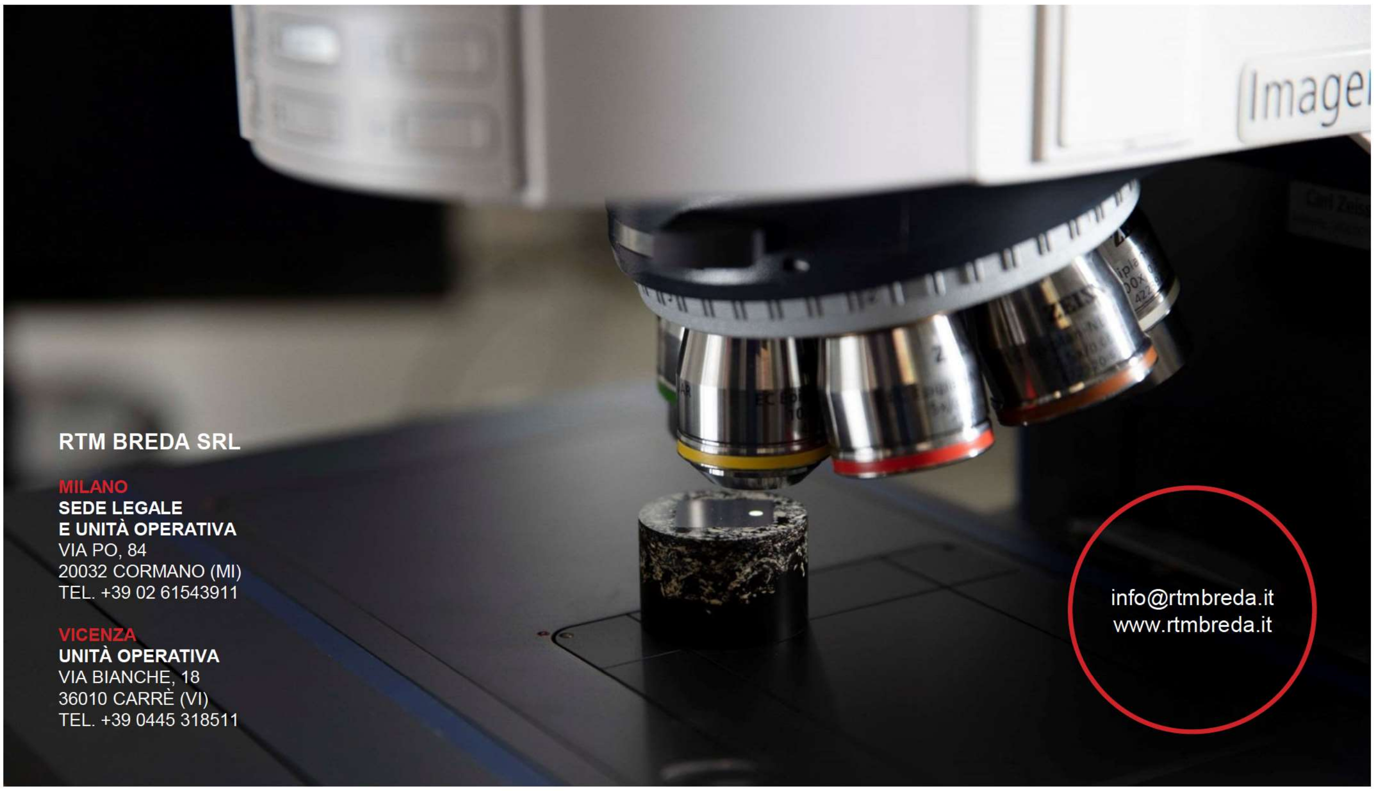

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