

Keywords: Back Stress Geometrically Necessary Dislocations Work Hardening Ductility Gradient Structure

Gradient structure in metals represents a new strategy for producing a superior combination of high strength and good ductility.[1-6] The gradient structure usually consists of a nanostructured (NS) surface layer with increasing grain size along the depth to reach coarse-grained (CG) sizes in the central layer.[2,4]

Gradient structure can promote ductility significantly,[2,4-9] which is measured under tensile loading. The NS layer in a gradient structure may sustain a large amount of tensile strain,[2,4] because they are constrained by the CG layer. It was reported that the gradient structured (GS) Cu derives its ductility from the confinement of the soft CG core,[2,10] and from strong grain growth in the NS layer by mechanically driven grain growth during tensile deformation. Nanostructures in high-purity copper are known to be unstable at room temperature, and mechanical-driven grain growth in

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The natu... fact, the... thin layers with increasing grain sizes.[3,4] The gradient structure deforms



heterogeneously due to plastic incompatibilities between neighboring layers with different flow behaviors and stresses under applied strains. As such, it is reasonable to anticipate the development of the strain gradient and internal stresses during plastic deformation, as a result of the plastic incompatibilities between different layers, similar to what happens in composites [19-21] and dual-phase structures.[22]

Back stress has been reported to play a crucial role in strain hardening, strengthening and mechanical properties.[21-23] It is a type of long-range stress exerted by GNDs that are accumulated and piled up against barriers. It interacts with mobile dislocations to affect their slip.[24] The back stress reduces the effective resolved shear stress for dislocation slip because it always acts in the opposite direction of the applied resolved shear stress. In a heterogeneous structure, strain will be inhomogeneous but continuous, producing strain gradients, which needs to be accommodated by GNDs. [23,25-27] It has been observed that back stress strengthening and back stress strain-hardening are primarily responsible for unprecedented combination of strength and ductility of heterogeneous lamella Ti, which was found as strong as ultrafine-grained Ti and as ductile as CG Ti.[23] The gradient structure can be regarded as a type of heterogeneous structure. Therefore, it is reasonable to assume that significant back stress will be developed in gradient structure, which should be investigated to have a better understanding on the fundamentals of gradient structure.

Here we report for the first time unambiguous experimental evidences of significant back stress hardening in GS IF steel. We will also derive an equation with sound physics to calculate back stress from an unloading-reloading stress-strain hysteresis loop during a

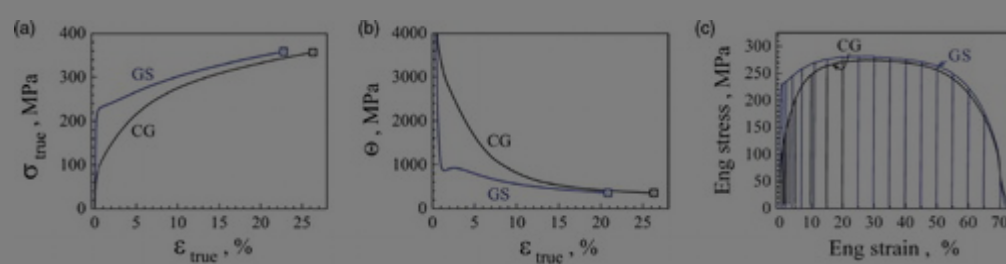
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Unloading-reloading process during tensile tests was conducted using an Instron 5966 machine at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ at room temperature. Tensile specimens with a gauge length of 10 mm and a width of 2.5 mm were cut from SMAT-processed disks. An extensometer was used to measure tensile strain. At a certain unloading strain, the specimen was unloaded in a load-control mode to 20 N at an unloading rate of 200 N min^{-1} , followed by reloading to the same applied load.

Figure 1(a) shows the monotonic tensile true stress-true strain (σ - ϵ) curves in both GS and CG samples. The GS sample shows large tensile ductility comparable to that of CG, but with triple yield strength of CG, which is typical of the excellent combination of strength and ductility in GS metals.[2-8] A transient is visible soon after yielding, characterized by the presence of a short concave segment on the σ - ϵ curve.[4] During the transient, the strain hardening rate (Θ) sharply drops at first, which is followed by a rapid up-turn, as shown in Figure 1(b). Figure 1(c) shows the unloading and reloading test hysteresis loops measured at varying tensile strains for both CG and GS samples.

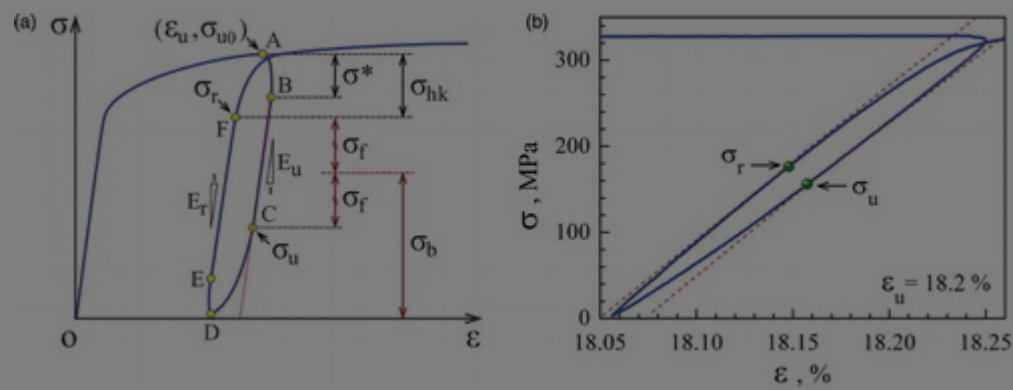
Figure 1. (Colour online) (a) Tensile stress-strain curves in the GS and CG IF steel samples. (b) Strain hardening rate ($\Theta = d\sigma/d\epsilon$) vs. strain. (c) The unloading and reloading test hysteresis loops measured at varying tensile strains for both CG and GS samples.



Unloading evolution of the unloading and reloading process starts at the quasi-elastic stage. The stress drop in the unloading curve [29] or reloading stress during unloading is an effective reloading Young's modulus of E_r , which can be assumed equal to E_u because the

microstructure is assumed not changed during the unloading-reloading. The point F is called the reloading yielding point, with a stress of σ_r . Figure 2(b) is the measured hysteresis loop from a GS IF steel sample.

Figure 2. (Colour online) (a) The schematic of the unloading-reloading loop for defining the unload yielding σ_u , reload yielding σ_r , back stress σ_b and frictional stress σ_f , effective unloading Young's modulus of E_u , effective reloading Young's modulus of E_r . (b) A measured hysteresis loop from the GS IF steel sample with σ_u and σ_r defined.



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From the unloading-reloading hysteresis loop, we can calculate the back stress σ_b , and the frictional stress σ_f . The back stress is always in the opposite direction of the applied stress, while frictional stress is always in the direction that opposes the motion of dislocations. The frictional stress consists of the Peierls stress as well as other stresses that are needed to overcome the dynamic pinning of dislocations such as solute atoms, second phase, forest dislocations, dislocation debris, dislocation jogs, etc.

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where σ_u is the unloading yield stress as defined in Figure 2(a).

During the reloading, the applied stress needs to overcome the back stress and the frictional stress to drive the dislocation forward at the reloading yield point F, which can be described as

$$(2)$$

where σ_r is the reloading yield stress as defined in Figure 2(a).

Here again, we assume that the back stress during reloading is the same as the back stress during unloading. This is reasonable because during the unloading-reloading process, dislocation configuration can be considered reversible.[32] Solving Equations (1) and (2) yields

$$(3)$$

and

$$(4)$$

Equation (3) is similar to an earlier equation proposed for cyclic loading by Cottrell [33] and Kulmann-Wilsdorf and Laird,[32] except they used σ_{u0} , the initial flow stress at the beginning of the unloading, in place of the σ_r , that is

$$(5)$$

where σ_{u0} is the initial unloading stress as defined in Figure 2(a).

We argue that Equation (3) is physically sounder than Equation (5) because we are defining unloading yield and reloading yield using the same criterion, that is, the same deviation of effective Young's modulus as discussed later. It has been recognized that Equation (5) overestimates the back stress, and was later modified by Dickson et al. to include the

where σ_r is the reloading yield stress as defined in Figure 2(a),[24,29] which is

Equation (3) is physically sounder than Equation (5) because we are defining unloading yield stress using the same criterion, that is, the same deviation of effective Young's modulus as discussed later. It has been recognized that Equation (5) overestimates the back stress, and was later modified by Dickson et al. to include the negative back stress. We expect that the back stress σ_u is important for the consistent back stress evolution. Physically



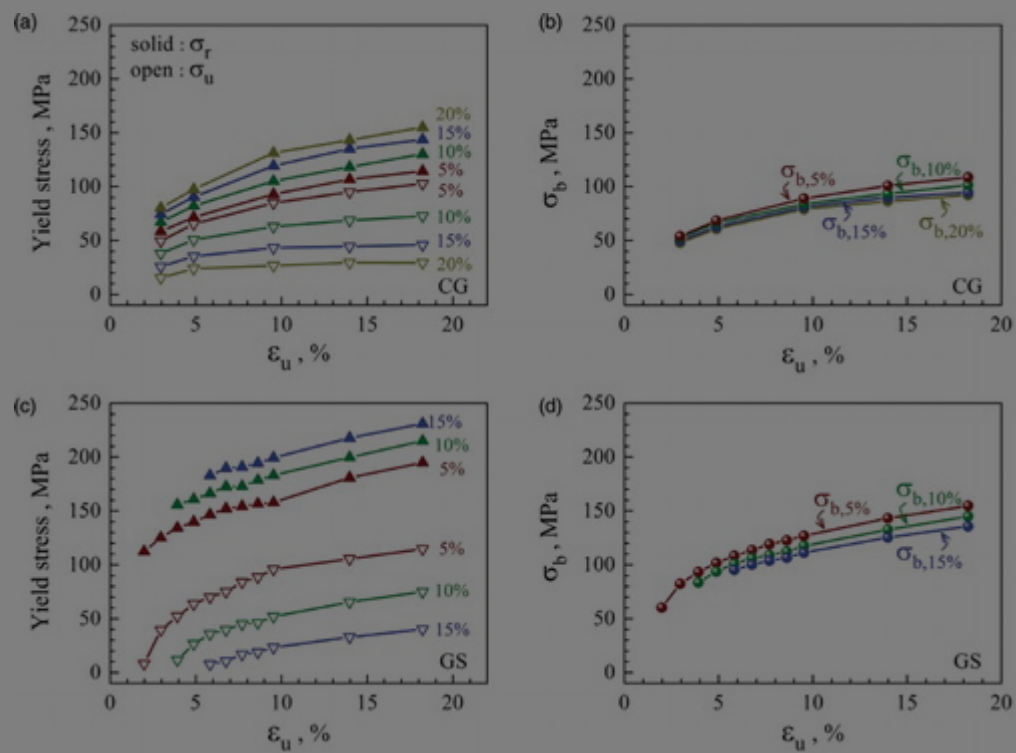
problematic because they implicitly used different criteria to define the unloading yield and reloading yield, which is physically unjustifiable.

To extract useful data from the unloading-reloading hysteresis loop, one needs to first determine the unloading yield stress σ_u and reloading yield stress σ_r . However, the real hysteresis loop (e.g. Figure 2(b)) is not as well defined as in Figure 2(a), and the practical extraction of the data is not straightforward.[31] The first step is to determine the elastic segments BC as well as its slope (the effective Young's modulus). The unloading yield point C is usually determined by a plastic strain offset in the range of 5×10^{-6} to 10^{-3} , which have been used by different research groups.[24,31,34-37] These offset values are arbitrary and are not well justified. Here we propose to use the deviation of the stress-strain slope from the effective Young's modulus as a physically sound method to determine the yield point. In this study, we choose 5%, 10%, and 15% slope reduction from the effective Young's modulus, E_u . If the strain hardening in the plastically deforming volume is ignored, the slope reduction should be equal to the volume fraction that is plastically deforming. For example, a 10% reduction in E_u means 10% of the sample volume is plastically deforming. We also propose to use $E_r = E_u$, and the same slope reduction values for determining both the unloading yield point and reloading yield point.

Figure 3 compares the evolution of the unloading yield stress, reloading yield stress, and back stress of the CG and GS IF steel samples with increasing tensile strain at which the unloading was initiated. Several features can be seen from the figure. First, the unloading yield stress is affected more than the reloading yield stress by the slope reduction using a 10% slope reduction. Second, higher reloading yield stress is observed for the choice of 10% slope reduction. The choice of 10% slope reduction is a better choice in the latter in the case of GS samples. The advantage of using 10% slope reduction is also reported in the literature for GS samples. The 5 sample stress in the case of 10% slope reduction is shown in Figure 3(b) and 3(d). The value is used, the

negative and therefore cannot be measured in the unloading curve. This makes it advantageous to use a smaller slope reduction value in determining the back stress.

Figure 3. (Colour online) Evolution of (a) unloading yield stress σ_u and reloading yield stress σ_r and (b) back stress with increasing unloading strain ϵ_u for CG IF steel, and the evolution of (c) unloading/reloading yield stresses and (d) back stress with increasing ϵ_u for GS IF steel. $\sigma_{b,5\%}$ represents the back stress calculated using 5% slope reduction from the effective Young's modulus.



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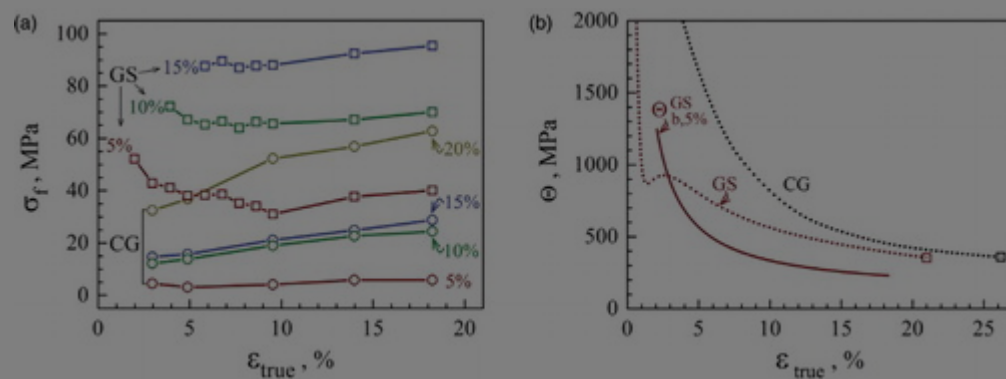
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Equation (4) is not quantitatively dependable. Nevertheless, Figure 4(a) consistently shows that for any slope reduction value, the calculated frictional stress is higher in the GS sample than in the CG sample. This is due to the higher dislocation density in the GS sample than in the CG sample.[3,4]

Figure 4. (Colour online) The frictional stress σ_f vs. tensile strain ϵ_{true} for the GS and CG IF steel samples calculated according to Equation (4). (b) The distinct back stress hardening in GS IF steel. denotes the back stress hardening rate calculated using 5% slope reduction from the effective Young's modulus.



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Figure 4(b) shows that the GS sample has much higher back stress strain-hardening than the CG sample due to the heterogeneous microstructure, especially in the transient range that correlates to Θ up-turn. This indicates that the back stress strain-hardening has significant contribution to the observed Θ up-turn. The rapid back stress increase right after the yielding of the GS sample is also obvious in Figure 3(d). The observed Θ up-turn has been attributed to fast dislocation accumulation due to the

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In summary, it is found that the GS IF steel developed strong back stress strengthening and back stress strain-hardening during tensile testing, which arise from the plastic incompatibilities due to its microstructural heterogeneity. The high back stress near the beginning of the plastic deformation of the GS IF steel samples should have contributed to the observed synergetic strengthening,[3] while the high back stress hardening should have contributed to the observed high ductility.[4] The equation derived and the procedure proposed in this work for calculating the back stress from the unloading-reloading hysteresis loop produces more consistent back stress value than what is previously reported.

Acknowledgements

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Disclosure statement

No potential conflict of interest was reported by authors.

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2. Fang T
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[PubMed](#) | [Web of Science ®](#) | [Google Scholar](#)

3. Wu XL, Jiang P, Chen L, et al. Synergetic strengthening by gradient structure. Mater Res Lett. 2014;2(4):185–191. doi: 10.1080/21663831.2014.935821

[Web of Science ®](#) | [Google Scholar](#)

4. Wu XL, Jiang P, Chen L, Yuan FP, Zhu YT. Extraordinary strain hardening by gradient structure. Proc Natl Acad Sci USA. 2014;111(20):7197–7201. doi: 10.1073/pnas.1324069111

[PubMed](#) | [Web of Science ®](#) | [Google Scholar](#)

5. Jerusalem A, Dickson W, Perez-Martin MJ, Dao M, Lu J, Galvez F, Grain size gradient length scale in ballistic properties optimization of functionally graded nanocrystalline steel plates. Scr Mater. 2013;69(11):773–776. doi: 10.1016/j.scriptamat.2013.08.025

[Web of Science ®](#) | [Google Scholar](#)

6. Wang HT, Tao NR, Lu K. Architected surface layer with a gradient nanotwinned structure in a Fe-Mn austenitic steel. Scr Mater. 2013;68(1):22–27. doi: 10.1016/j.scriptamat.2012.05.041

[Web of Science ®](#) | [Google Scholar](#)

7. Kou HN, Lu L, Li Y. High-strength and high-ductility nanostructured and amorphous metal

10.10

8. Wei YJ, et al. High strength and high ductility in steel with a gradient nanostructure. Mater Res Lett. 2014;2(4):185–191. doi: 10.1080/21663831.2014.935821



9. Ma XL, et al. High strength and high ductility in a steel with a gradient nanostructure. Mater Res Lett. 2014;2(4):185–191. doi: 10.1080/21663831.2014.935821

10.10

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0. Fang TH, Tao NR, Lu K. Tension-induced softening and hardening in gradient nanograined surface layer in copper. *Scr Mater.* 2014;**77**:17-20. doi: 10.1016/j.scriptamat.2014.01.006

 | [Web of Science ®](#) | [Google Scholar](#)

1. Weertman JR. Retaining the nano in nanocrystalline alloys. *Science.* 2012;**337**(6097):921-922. doi: 10.1126/science.1226724

 | [PubMed](#) | [Web of Science ®](#) | [Google Scholar](#)

2. Chookajorn T, Murdoch HA, Schuh CA. Design of stable nanocrystalline alloys. *Science.* 2012;**337**(6097):951-954. doi: 10.1126/science.1224737

 | [PubMed](#) | [Web of Science ®](#) | [Google Scholar](#)

3. Zhang K, Weertman JR, Eastman JA. Rapid stress-driven grain coarsening in nanocrystalline Cu at ambient and cryogenic temperatures. *Appl Phys Lett.* 2005;**87**(6): Article no. 061921. doi: 10.1063/1.2008377

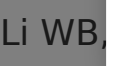
 | [Web of Science ®](#) | [Google Scholar](#)

4. Zhang K, Weertman JR, Eastman JA. The influence of time, temperature, and grain size on indentation creep in high-purity nanocrystalline and ultrafine grain copper. *Appl Phys Lett.* 2004;**85**:5197-5199. doi: 10.1063/1.1828213

 | [Web of Science ®](#) | [Google Scholar](#)

5. Legros  | [Web of Science ®](#) | [Google Scholar](#) boundary motion. *Appl Phys Lett.* 2014;**104**(14):3380-3393.

6. Lia  | [Web of Science ®](#) | [Google Scholar](#) JF, Zhu YT. High strength and ductility of nanocrystalline Ni. *Appl Phys Lett.* 2014;**104**(14):3380-3393.

7. Li WB,  | [Web of Science ®](#) | [Google Scholar](#) with gradient nano-grained structure. *AIP Advances.* 2015;**5**(8): Article no. 087120. doi: 10.1063/1.497120



10.1063/1.4928448

[Google Scholar](#)

8. Li JJ, Chen SH, Wu XL, Soh AK. A physical model revealing strong strain hardening in nano-grained metals induced by grain size gradient structure. *Mater Sci Eng A*. 2015;**620**:16–21. doi: 10.1016/j.msea.2014.09.117

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9. Llorca J, Needleman A, Suresh S. The Bauschinger effect in whisker-reinforced metal-matrix composites. *Scr Metall Mater*. 1990;**24**(7):1203–1208. doi: 10.1016/0956-716X(90)90328-E

[Google Scholar](#)

10. Sinclair CW, Saada G, Embury JD. Role of internal stresses in co-deformed two-phase materials. *Philos Mag*. 2006;**86**(25–26):4081–4098. doi: 10.1080/14786430600654438

[Web of Science](#) [®] | [Google Scholar](#)

11. Thilly L, Van Petegem S, Renault PO, Lecouturier F, Vidal V, Schmitt B, Van Swygenhoven H. A new criterion for elasto-plastic transition in nanomaterials: application to size and composite effects on Cu-Nb nanocomposite wires. *Acta Mater*. 2009;**57**(11):3157–3169. doi: 10.1016/j.actamat.2009.03.021

[Web of Science](#) [®] | [Google Scholar](#)

12. Calca... mechanisms in
fine- a... effect of
aging...

13. Wu... us lamella
struct... Natl Acad Sci
USA. 2



24. Feaugas X. On the origin of the tensile flow stress in the stainless steel AISI 316L at 300 K: back stress and effective stress. *Acta Mater.* 1999;**47(13)**:3617–3632. doi: 10.1016/S1359-6454(99)00222-0

 | [Web of Science®](#) | [Google Scholar](#)

25. Ashby MF. The deformation of plastically non-homogeneous materials. *Philos Mag.* 1970;**21(170)**:399–424. doi: 10.1080/14786437008238426

 | [Web of Science®](#) | [Google Scholar](#)

26. Gao H, Huang Y, Nix WD, Hutchinson JW. Mechanism-based strain gradient plasticity - I Theory. *J Mech Phys Solids.* 1999;**47(6)**:1239–1263. doi: 10.1016/S0022-5096(98)00103-3

 | [Web of Science®](#) | [Google Scholar](#)

27. Gao HJ, Huang YG. Geometrically necessary dislocation and size-dependent plasticity. *Scr Mater.* 2003;**48(2)**:113–118. doi: 10.1016/S1359-6462(02)00329-9

 | [Web of Science®](#) | [Google Scholar](#)

28. Lu K, Lu J. Nanostructured surface layer on metallic materials induced by surface mechanical attrition treatment. *Mater Sci Eng A.* 2004;**375–377**:38–45. doi: 10.1016/j.msea.2003.10.261

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29. Dickson... measuring...
cyclic... . doi:
10.10...

30. Fo... of the
hys... creep and
stress... ;437:197-
211. o



1. Fournier B, Sauzay M, Caes C, Noblecourt M, Mottot M. Analysis of the hysteresis loops of a martensitic steel - Part I: study of the influence of strain amplitude and temperature under pure fatigue loadings using an enhanced stress partitioning method. Mater Sci Eng A. 2006;**437**:183-196. doi: 10.1016/j.msea.2006.08.086

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2. Kuhlmann-Wilsdorf D, Laird C. Dislocation behavior in fatigue II. Friction stress and back stress as inferred from an analysis of hysteresis loops. Mater Sci Eng. 1979;**37**(2):111-120. doi: 10.1016/0025-5416(79)90074-0

 | [Google Scholar](#)

3. Cottrell AH. Dislocations and plastic flow in crystals. Oxford: Clarendon Press; 1953.

[Google Scholar](#)

4. Delobelle P, Oytana C. The study of the laws of behavior at high-temperature, in plasticity-flow, of an austenitic stainless-steel (17-12-Sph). J Nucl Mater. 1986;**139**(3):204-227. doi: 10.1016/0022-3115(86)90174-1

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5. Risbet M, Feugas X, Clavel M. Study of the cyclic softening of an under-aged gamma'-precipitated nickel-base superalloy (Waspaloy). Journal De Physique IV. 2001;**11**(PR4):293-301. doi: 10.1051/jp4:2001436

 | [Google Scholar](#)

36. Guilleminot A, Lecomte J, Lecomte J, Lecomte J. Kinetics in nodular cast iron. Mater Sci Eng. 2000;**33**:1-10. doi: 10.1016/S0921-5097(00)00001-1

37. Morin G, Lecomte J, Lecomte J, Lecomte J. Strain rate effect on the fatigue crack growth kinetics in nodular cast iron. Mater Sci Eng. 2004;**45**:24-30. doi: 10.1016/j.msea.2004.08.001



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