Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat



Extraordinary Bauschinger effect in gradient structured copper

Xiaolong Liu^a, Fuping Yuan^{a,b}, Yuntian Zhu^{c,d}, Xiaolei Wu^{a,b,*}

^a State Key Laboratory of Nonlinear Mechanics, Institute of Mechanics, Chinese Academy of Sciences, 15 Beisihuan West Road, Beijing 100190, China

^b College of Engineering Sciences, University of Chinese Academy of Sciences, Yuquan Road, Beijing 100049, China

^c Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695, USA

^d School of Materials Science and Engineering, Nanjing University of Science and Technology, Nanjing 210094, China

ARTICLE INFO

Article history: Received 24 October 2017 Received in revised form 5 March 2018 Accepted 6 March 2018 Available online 20 March 2018

Keywords: Bauschinger effect Geometrically necessary dislocations Gradient structure Reverse yield softening Tensile-compressive test

ABSTRACT

Bauschinger effect is a well-known phenomenon, in which the tensile stress is higher than the reverse compressive stress. Here we report that the gradient structured copper exhibits an extraordinarily large Bauschinger effect. We propose to use the reverse yield softening, $\Delta \sigma_b$, as a quantitative parameter to represent the Bauschinger effect. $\Delta \sigma_b$ evolves in the same trend as the back stress with pre-strain, and can be used to evaluate the effectiveness of a heterostructure in producing back stress for superior mechanical properties.

© 2018 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

The Bauschinger effect has been extensively reported in conventional homogenous materials [1–3]. However, the Bauschinger effect in homogenous materials is usually not very strong, and has not been related with mechanical properties such as strength and ductility. Recently, the heterostructured metallic materials have been found to have both strong Bauschinger effect and high back stress [4]. The gradient structure can be considered a type of heterogeneous structure [5], and was also reported to have high back stress [6]. In addition, back stress hardening was found, i.e. the back stress increases with preapplied tensile strain (pre-strain). These reports raise two interesting issues: 1) How does the Bauschinger effect evolve with pre-strain in a heterostructure? 2) Can the Bauschinger effect be quantified and related to the mechanical properties such as strength and ductility?

The gradient structure is characterized with by increasing grain size along the depth from the nanostructured (NS) surface layer to coarsegrained (CG) central layer. It has been found to suppress the strain localization and represents a new strategy for producing a superior combination of high strength and good ductility [7,8]. The superior properties of gradient structured (GS) interstitial-free (IF) steel were attributed to extraordinary high strain hardening [9,10], a significant part of which is attributed to the back-stress hardening [4–6]. For example, it was found that the GS IF steel developed high back stress to increase its strength and high back-stress work hardening to maintain good ductility [6]. In other words, the back stress plays a major role in the mechanical behavior of gradient materials. It will be of interest to probe if the

* Corresponding author.

E-mail address: xlwu@imech.ac.cn (X. Wu).

https://doi.org/10.1016/j.scriptamat.2018.03.007

1359-6462/© 2018 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Bauschinger effect is related to superior mechanical properties in gradient materials.

Strain hardening is the primary approach for improving ductility [11]. There are two mechanisms to produce strain hardening in metals, i.e., forest dislocation hardening and back stress hardening [12–15]. The forest dislocation hardening is attributed to the accumulation of dislocations. The flow stress of a metal is described by [16,17]:

$$\tau = \alpha G b \sqrt{\rho_{\rm S} + \rho_{\rm G}} \tag{1}$$

where τ is the shear flow stress, α is a constant, *G* is the shear modulus, *b* is the magnitude of Burgers vector, ρ_S is the density of statistically stored dislocations (SSDs), ρ_G is the density of geometrically necessary dislocations (GNDs). Therefore, strain hardening caused by dislocation accumulation can be derived as

$$\frac{\mathrm{d}\tau}{\mathrm{d}\varepsilon} = \alpha G b \frac{\mathrm{d}\sqrt{\rho_{\mathrm{S}} + \rho_{\mathrm{G}}}}{\mathrm{d}\varepsilon} \tag{2}$$

where ε is the applied strain. Eq. (2) indicates the strain hardening is caused by the increase of total dislocation density with strain.

For the conventional homogeneous metals, dislocation accumulation is often the primary mechanism for strain hardening [18], for which Eqs. (1) and (2) can reasonably explain their mechanical behaviors. Back stress is rarely included in the explanation of the mechanical properties, because it is much weaker than forest dislocation hardening [5]. Bauschinger effect is usually not related to the mechanical properties either. However, for heterostructured metals, the back stress





contributes much more to work hardening than the forest dislocations do, and the Bauschinger effect is also much stronger [4]. Back stress hardening is caused by the pile-ups of GNDs [16,17], which are needed to accommodate strain gradient near the interfaces when strain partitioning occurs between the hard and soft domains [19–24]. For example, the excellent strain hardening capacity of heterostructured materials, such as nano-composites [25,26], precipitation alloys [27,28], and passivated films [29] is attributed to heterogeneous deformation between the soft and hard phases, which trigger the back stress hardening.

It is the objective of this work to investigate the evolution of Bauschinger effect with pre-strain, to quantify the Bauschinger effect and to relate the Bauschinger effect with the mechanical properties of heterostructured materials. Gradient structured (GS) Cu is used as for the experimental study, which was found to have an extraordinarily high Bauschinger effect due to its high back stress, because both Bauschinger effect and the back stress have the same physical origin.

Oxygen-free copper with the composition (wt%): P, 0.002%, Fe, 0.004%, Ni, 0.002%, Sn, 0.002%, S, 0.004%, Zn, 0.003%, O, 0.03% was used in this study. A copper rod of 12 mm in diameter was cut and annealed in vacuum at 873 K for 2 h to obtain homogeneous CG microstructure (Fig. 1a) with a mean grain size of 78 μ m. Test specimens are machined into a dog-bone shape with a gauge diameter of 3 mm and gauge length of 15 mm from the annealed bar. Some specimens were processed by means of surface mechanical attrition treatment (SMAT) to form a GS surface layer in the gauge section.

The SMAT duration was 30 min for each specimen. The GS layer of 250 μ m thick was formed, in which the grain size increases gradually with an increasing depth (Fig. 1b). In topmost 20 μ m thick layer the transversal grain sizes increase from 200 nm to 400 nm along the depth as shown in Fig. 1c and d (the location is framed by a box in

Fig. 1b). Fig. 1e presents the variation of average transversal grain sizes along depth from the surface. The original coarse grains are below the 250-µm depth.

Mechanical tests including the tensile tests and Bauschinger tensilecompressive tests were conducted using a MTS Landmark machine at strain rate of 5×10^{-4} s⁻¹ under strain control mode. The strain is measured with an extensometer. Full Bauschinger tensile-compressive tests were performed as schematically presented in Fig. 2a. Test programs were developed to conduct the Bauschinger tensile-compressive tests, i.e. a specimen is stretched to the certain strain (O-A-B in Fig. 2a) under strain control mode, and then unloaded and compressed beyond reverse yielding (B-C in Fig. 2a). Ten samples were used For CG-Cu, including one for monotonous tensile loading and nine for tensilecompressive tests (forward loading pre-strains are 2%, 4%, 6%, 8%, 10%, 12%, 15%, 20% and 25%). Seven samples of GS-Cu were used in the tests, including one for monotonous tensile loading and six for tensilecompressive loading (pre-strains are 2%, 4%, 6%, 8%, 10% and 15%). It is worth noting that no buckling occurred in our tests, mainly because the compressive strain was only about 2% in our tests, and the reverse yielding strength was measured at a strain offset of 0.2%. The initial yield strength σ_v , flow stress σ_f (marked by circle in Fig. 2a), reverse yield strength σ_{ry} (marked by square in Fig. 2a) can be obtained from tensile-compressive curves. It was proposed that back stress $\sigma_{\rm b}$ can be determined by the following equations [8]:

$$\sigma_{\rm b} = \frac{\sigma_{\rm f} + \sigma_{\rm ry}}{2} \tag{3}$$

However, the configuration of GNDs at the 0.2% reverse compressive strain is expected to be different from that at the beginning of



Fig. 1. Microstructure of CG-Cu and GS-Cu, (a) CG-Cu, (b) GS-Cu, (c) EBSD image and (d) grain size distribution for the topmost 3 µm thick layer of GS-Cu, and (e) variation of average transversal grain sizes along depth.



Fig. 2. (a) Schematic of the Bauschinger tensile-compressive tests, tensile curves and tensile-compressive curves for CG-Cu (b) and GS-Cu (c), and (d) reverse yield softening vs. pre-strain.

unloading. In other words, the configuration of GNDs would have been at least partially changed at such a large reverse strain. Consequently, the back stress measured using Eq. (3) would be lower than the real back stress at the beginning of unloading. Therefore, the back stress calculated using Eq. (3) can be considered as the lower bound of the back stress. More accurate measurement of back stress can be measured using an unloading-reloading procedure [6].

Bauschinger effect is a phenomenon that the reverse compressive yield stress is lower than the initial forward flow stress at the beginning of unloading. Therefore, it is reasonable to use the difference between the forward flow stress at the beginning of the unloading and the reverse compressive yield stress at 0.2% offset strain, i.e.

$$\Delta \sigma_{\rm b} = \sigma_{\rm f} - \left| \sigma_{\rm ry} \right| = \sigma_{\rm f} + \sigma_{\rm ry} \tag{4}$$

Here we define $\Delta\sigma_b$ as the Bauschinger reverse yield softening and propose to use $\Delta\sigma_b$ as a quantitative measure of the Bauschinger effect. Obviously, the value of $\Delta\sigma_b$ is twice the value of the lower-bound back stress calculated using Eq. (3). Namely, $\Delta\sigma_b$ can be related to the magnitude of back stress. Since the back stress has been used as an indicator of the effectiveness of a heterostructure in producing a superior combination of strength and ductility, the $\Delta\sigma_b$ can be also used to evaluate and optimize the heterostructure.

The fundamental basis for the above co-relationship between the Bauschinger effect and back stress is that they are both produced by the same physical phenomenon: the piling up of GNDs. Several other parameters have also been used to represent the Bauschinger effect, including Bauschinger strain and stress parameter [3] and permanent softening [30,31]. The Bauschinger reverse yield softening, $\Delta \sigma_b$ proposed here, has the advantage of clear physical definition, easy to measure, and can effectively relates the Bauschinger effect with mechanical behavior and properties of heterostructured materials. The higher Bauschinger effect, as quantitatively represented by $\Delta \sigma_b$, indicates a more effective heterostructure for superior mechanical properties.

Fig. 2b and c present the monotonic tensile true stress-strain curves and the tensile-compressive curves for CG-Cu and GS-Cu. The GS-Cu has excellent combination of high strength and good ductility. It is worth noting that no transient response in tensile curve of GS-Cu was observed, which was reported in other round bar GS specimens [7,32]. The differences in the tensile parts of the tensile-compressive curves among different specimens in each group are very small and negligible. As shown in Fig. 2b, for the homogeneous CG copper both the forward stress and reverse yield stress (marked by circle and square in Fig. 2b) increase with increasing applied strain, which is typical of isotropic hardening or forest dislocation hardening. This is because the dislocation density typically increases with increasing plastic strain. In contrast, Fig. 2c shows that for GS-Cu with the increasing applied tensile strain, the forward yield strength (marked by circles) increases, while the magnitude of the reverse yield strength (marked by squares) decreases. This is extraordinary and very different from behavior of the homogeneous CG sample. This is because the GS sample has very strong directional back stress, which causes extraordinarily high Bauschinger effect.

Fig. 2d shows the evolution of the reverse yield softening $\Delta \sigma_b$ with increasing applied forward tensile pre-strain. As shown, the GS Cu has a much stronger Bauschinger effect than its homogeneous CG counterpart. This means that the GS Cu has much higher back stress work hardening to help it with enhancing the strength and retaining the ductility.

The microstructure of CG-Cu and GS-Cu is totally different as shown in Fig. 1. The CG-Cu has a nearly homogeneous CG microstructure where plastic deformation is homogeneous. SSDs accumulate during homogeneous plastic straining. Their accumulation is mostly the result of chance encounters, which led leads to mutual trapping and dislocation accumulation [16]. The strain hardening caused by such dislocation density increase can be described by Eq. (2), and is non-directional. It should affect the forward flow stress and reverse yield stress the same way and therefore does not cause Bauschinger effect. However, even homogenous CG copper usually has some heterogeneity, which produces some Bauschinger effect with lower magnitude residual back stress as compared to the GS-Cu, as shown in Fig. 2d.

The microstructure of GS-Cu is heterogeneous and the grain sizes vary from nanoscale to microscale. Layers with different grain sizes have different flow stresses, which first leads to the development of two dynamically migrating plastic/elastic interfaces, and later two migrating necking/stable interfaces [9]. High strain gradient will be developed near the interfaces, which need to be accommodated by the GNDs. The accumulation of GNDs will cause a directional long-range back stress, which impedes dislocation slip in the tensile direction, and promotes dislocation slip in the reverse direction. Correspondingly, the tensile-compressive curves show a high forward flow stress and a reduced reverse yield stress as shown in Fig. 2c. In other words, this leads to both large Bauschinger effect and high back stress.

In summary, the gradient structured copper shows extraordinary Bauschinger effect in which the reverse yield stress increases with increasing pre-strain. This was caused by the large Bauschinger effect, as represented quantitatively by the large Bauschinger reverse yield softening, $\Delta\sigma_{\rm b}$, which is closely related with the back stress because they both have the same physical origin: piling up of GNDs. $\Delta\sigma_{\rm b}$ can be used as an effective parameter to quantitatively represent the magnitude of the Bauschinger effect. It can also be used as a parameter to evaluate the effectiveness of heterostructures in producing superior mechanical properties. With the larger pre-strain, the Bauschinger effect increases faster in GS-Cu than in CG-Cu, indicating high backstress hardening in the GS Cu.

Acknowledgements

This work was supported by National Key R&D Program of China (2017YFA0204402), and the National Natural Science Foundation of China under Grant Nos. 11572328, 11472286, 11672313, 11790293. The Strategic Priority Research Program of the Chinese Academy of Sciences under Grant No. XDB22040503. YTZ acknowledges the support of the US Army Research Office (W911NF-17-1-0350).

References

- [1] G.D. Moan, J.D. Embury, Acta Metall. 27 (1979) 903.
- T. Kishi, I. Gokyu, Metall. Mater. Trans. B Process Metall. Mater. Process. Sci. 4 (1973) 390.
- [3] A. Abel, H. Muir, Philos. Mag. 26 (1972) 489.
- [4] X.L. Wu, M.X. Yang, F.P. Yuan, G.L. Wu, Y.J. Wei, X.X. Huang, Y.T. Zhu, Proc. Natl. Acad. Sci. U. S. A. 112 (2015) 14501.
- [5] X.L. Wu, Y.T. Zhu, Mater. Res. Lett. 5 (2017) 527.
- [6] M.X. Yang, Y. Pan, F.P. Yuan, Y.T. Zhu, X.L. Wu, Mater. Res. Lett. 1 (2016) 1.
- [7] T.H. Fang, W.L. Li, N.R. Tao, K. Lu, Science 331 (2011) 1587.
- [8] K. Lu, Nat. Rev. Mater. 1 (2016) 16019.
- [9] X.L. Wu, P. Jiang, L. Chen, F.P. Yuan, Y.T. Zhu, Proc. Natl. Acad. Sci. U. S. A. 111 (2014) 7197.
- [10] X.L. Wu, P. Jiang, L. Chen, Y.T. Zhu, Mater. Res. Lett. 2 (2014) 185.
- [11] R.Z. Valiev, Y. Estrin, Z. Horita, T.G. Langdon, M.J. Zehetbauer, Y.T. Zhu, Mater. Res. Lett. 4 (2016) 1.
- [12] K.M.J. Akhtar, S. Khan, Int. J. Plast. 15 (1999) 1265.
- [13] X. Feaugas, Acta Mater. 47 (1999) 3617.
- [14] G. Fribourg, Y. Bréchet, A. Deschamps, A. Simar, Acta Mater. 59 (2011) 3621.
- [15] K. Maciejewski, H. Ghonem, Int. J. Fatigue 68 (2014) 123.
- [16] M.F. Ashby, Philos. Mag. 21 (1970) 399.
- [17] H. Gao, Y. Huang, W.D. Nix, J.W. Hutchinson, J. Mech. Phys. Sol. 47 (1999) 1239.
 [18] Y.T. Zhu, X.Z. Liao, X.L. Wu, Prog. Mater. Sci. 57 (2012) 1
- [18] Y.T. Zhu, X.Z. Liao, X.L. Wu, Prog. Mater. Sci. 57 (2012) 1.
 [19] Z. Sun, G. Song, T.A. Sisneros, B. Clausen, C. Pu, L. Li, Y. Gao, P.K. Liaw, Sci. Rep. 6 (2016) 1.
- [20] J. Gilsevillano, Scr. Mater. 60 (2009) 336.
- [21] D.S. Yan, C.C. Tasan, D. Raabe, Acta Mater. 96 (2015) 399.
- [22] J.J. Li, S.H. Chen, X.L. Wu, A.K. Soh, Mater. Sci. Eng. A 620 (2015) 16.
- [23] J.J. Li, G.J. Weng, S.H. Chen, X.L. Wu, Int. J. Plast. 88 (2017) 89.
- [24] W.B. Li, F.P. Yuan, X.L. Wu, AIP Adv. 5 (2015), 087120.
- [25] G. Badinier, C.W. Sinclair, S. Allain, O. Bouaziz, Mater. Sci. Eng. A 597 (2014) 10.
- [26] L. Thilly, S.V. Petegem, P.O. Renault, F. Lecouturier, V. Vidal, B. Schmitt, H.V. Swygenhoven, Acta Mater. 57 (2009) 3157.
- [27] A. Reynolds, J. Lyons, Metal. Mater. Trans. A 28 (1997) 1205.
- [28] M.X. Yang, F.P. Yuan, Q.G. Xie, Y.D. Wang, E. Ma, X.L. Wu, Acta Mater. 109 (2016) 213.
- [29] Y. Xiang, J.J. Vlassak, Scr. Mater. 53 (2005) 177.
- [30] B.K. Chun, J.T. Jinn, J.K. Lee, Int. J. Plast. 18 (2002) 571.
- [31] M.P. Miller, E.J. Harley, D.J. Bammann, Int. J. Plast. 15 (1999) 93.
- [32] H.W. Huang, Z.B. Wang, J. Lu, K. Lu, Acta Mater. 87 (2015) 150.