Materials Research Express

PAPER

Effect of bimodal grain size and gradient structure on heterogeneous deformation induced (HDI) stress and mechanical properties of Cu

Yi Yang¹, Yulan Gong¹, Xingfu Li¹, Huan Liu¹, Cong Li¹, Jingran Yang¹, Hongjiang Pan¹, Baipo Shu¹, Chao Deng¹, Junqi Fang¹ and Xinkun Zhu¹

¹ Faculty of Materials Science and Engineering, Kunming University of Science and Technology, Kunming, Yunnan, 650093, People’s Republic of China
² Faculty of Science, Kunming University of Science and Technology, Kunming, Yunnan, 650093, People’s Republic of China
³ Faculty of Materials Science and Engineering, Chongqing University, Chongqing, 400044, People’s Republic of China
⁴ Shimadzu management China Co, No. 230 Gaotang Road, Tianhe District, Guangzhou, Ltd, People’s Republic of China

E-mail: sk_zhu@hotmail.com

Keywords: surface mechanical attrition treatment (smat), bimodal grain size structure (bgss), gradient structure (gs), heterogeneous deformation induced (hdi) stress, copper

Abstract

In this study, two different types of heterogeneous structures were prepared by controlling different surface mechanical attrition treatment (SMAT) time (5 s, 10 s, 30 s, 60 s). The effect of heterogeneous Cu with bimodal grain size structure (BGSS) and gradient structure (GS) on heterogeneous deformation induced (HDI) stress and mechanical properties was investigated systematically. By combining surface morphology with microhardness distribution, it was found that the surface grains of the SMAT-ed Cu (5 s and 10 s) were partially refined to exhibit BGSS, while the surface grains of SMAT-ed Cu (30 s and 60 s) were sufficiently refined to form GS. The load-unload-reload (LUR) tests results showed that HDI stress for SMAT 60s Cu was much higher than that of the SMAT 10s Cu. Furthermore, microstructure characterizations revealed that SMAT-ed Cu with BGSS and GS suppressed strain localization, which resulted in high strength and reasonable ductility.

1. Introduction

Nanostructured (NS) materials have attracted attention due to their excellent mechanical properties [1, 2]. In general, NS materials were prepared by severe plastic deformation (SPD), such as high-pressure torsion (HPT), accumulative roll-bonding (ARB) and equal-channel angular pressing (ECAP) [3–8]. However, NS materials exhibit the trade-off between strength and ductility, i.e., high strength and limited ductility [9]. The low ductility of NS materials was attributed to lack of strain hardening [10–13]. Therefore, it was crucial to explore the underlying mechanisms of enhanced yield strength and reasonable ductility.

Recently, it has been reported that heterogeneous materials exhibit high strength while maintaining ductility [14–18]. For example, Ma et al [19] reported that Cu with the bimodal grain size distribution exhibited strength-ductility synergy. The underlying mechanism is that NS provides high strength, while larger grains can generate strain hardening. From a gradient structure (GS) perspective, Lu et al [20] reported that GS Cu possessed extraordinary strength and ductility, which resulted from nanograins growth. In addition, Zha et al [21] has proposed that the heterogeneous lamella structure was produced by asymmetric rolling and subsequent partial recrystallization, which showed outstanding property combination originating from geometrically necessary dislocations (GNDs) generation and accumulation, resulting in heterogeneous deformation induced (HDI) stress strengthening and HDI strain hardening. These materials have one common feature. The substantial differences in strength between the different domains have a significant influence on the strength and ductility of the material. Surface mechanical attrition treatment (SMAT) has been reported to produce a gradient structure (GS), which can increase the strength of the material without noticeably reducing ductility [22–24]. In addition, Lu et al [25] has described the mechanisms of planar heterogeneous structures in the SMAT-ed materials in term
of finite element simulations and theoretical modelling. However, effect of bimodal grain size and gradient structure on HDI stress and mechanical properties in the SMAT-ed Cu has not been investigated systematically.

In this study, two different heterogeneous structured materials were prepared by SMAT at room temperature for different time. The load-unload-reload (LUR) tests were performed to measure HDI stress of heterogeneous Cu with bimodal grain size and gradient structure. Including digital image correlation (DIC), transmission electron microscope (TEM) and electron back scatter diffraction (EBSD), various microstructural characterizations were employed to investigate effect of Cu with different heterostructures on HDI stress and mechanical properties.

2. Experimental

Pure Cu (99.95%) was rolled into sheets with 1 mm thickness and then annealed at 973 K for 2 h in vacuum to obtain homogeneous coarse grains (CG). The SMAT device (as shown in figure 1) was composed of a vibration generator and a processing chamber holding the steel balls as well as the sample. Both sides of Cu were subjected to SMAT under different time (5 s, 10 s, 30 s, 60 s). These SMAT-ed Cu were labeled as SMAT 5s Cu, SMAT 10s Cu, SMAT 30s Cu and SMAT 60s Cu, respectively. The SMAT processing was described in previous studies [26, 27].

After SMAT, nano-indentation with force of 2500 μN was tested to measure cross-sectional hardness distribution of the SMAT 5s Cu sample. Vickers microhardness experiment was also conducted at intervals of 25 μm with the force of 0.098 N and the dwell time of 15 s, the hardness value for each depth was the average of 5 indentations. The quasi-static uniaxial tensile samples were cut into dog-bone shape (50 mm × 10 mm × 1 mm). Tensile tests were carried out on a Shimadzu AG-X universal material machine with a strain rate of 5 × 10^{-4} s^{-1} at room temperature. In addition, load-unload-reload (LUR) tests were conducted with a loading speed of 0.375 mm min^{-1} and an unloading speed of 1000 N min^{-1}. Three tensile tests were carried out on each sample under the same test conditions to ensure repeatability of the data.

In-situ DIC test with CCD camera was used to record the strain evolution on the surface of the SMAT samples during the tensile strain. The DIC samples were prepared by spraying white paint followed by spraying randomly distributed black spots on the surface of the samples. The strain evolution of the SMAT samples were further analyzed by the software (GOM Correlate). Additionally, the surface roughness of the SMAT 10s and SMAT 60s Cu before tensile and after tensile to a strain of 2% were measured by a 3D white light interferometric surface topography (Zygo Nexview). To observe the cross-section microstructure of the SMAT-ed Cu, field emission scanning electron microscope (FE-SEM) was utilized with an electron back scatter diffraction (EBSD) detector. Microstructural characterization was carried out under a transmission electron microscope (TEM, FEI Tecnai-F80) with the voltage of 200 KV at a depth of 70 μm from the SMAT-ed surface. The samples were polished with sandpaper from one side to approximately 70 μm and then double-sprayed with a solution (V (phosphoric acid): V (alcohol): V (water) = 1:1:2) at 24 °C.
3. Results and discussion

3.1. Surface morphology and cross-sectional microhardness

Different surface morphologies of SMAT-ed Cu can be observed by controlling different SMAT time, because the surface was randomly shocked by the steel balls during the SMAT process, as shown in figure 2. The deformed zones and non-deformed zones of the sample surface were defined as hard domains and soft domains, respectively. The hard domains of the SMAT 5s and SMAT 10s Cu accounted for 16.67% and 71.84% of the total surface, respectively. With increasing SMAT time, the SMAT 30s and SMAT 60s Cu were fully covered by the hard domains.

Nano-indentation result below the hard domains of the SMAT 5s Cu is present in figure 3(a). It is found that the hardness decreases from the surface to the center, and the hardness varies over a range of 25 μm with an average hardness of 1.49 Gpa. From figure 2, partial hard domains were formed. Therefore, the processing in this condition can be considered as randomly distributed hard domains embedding into soft domains to generate a bimodal grain size structure (BGSS). In addition, Vickers microhardness was carried out for the SMAT 30s Cu. The hardness of the topmost surface is the highest from figure 3(b), which exhibits 1.18 times higher than that of the inner layer. As the cross-sectional depth increases, the hardness gradually decreases until it remains almost unchanged at the depth of 300 μm. It can be found that hardness variation with 300 μm thickness for the SMAT 30s Cu is higher than that of the SMAT 5s Cu with 25 μm thickness due to increasing SMAT time. It suggests that the volume fraction of the hard domains of the SMAT 30s Cu is higher than that of the SMAT 5s Cu. Combining with surface morphology and microhardness distribution, the surface of SMAT 5s and SMAT 10s Cu were marked as BGSS, whereas the surface of SMAT 30s and SMAT 60s Cu were labeled as gradient structure (GS). Hardness gradient variation can be attributed to gradient strain in the SMAT-ed Cu. The topmost surface of samples had the highest strain, the surface grains was refined and their hardness increased significantly.

3.2. Microstructure characterization

3.2.1. EBSD characterization

In figure 4(a), the image quality (IQ) maps show that there is no grain size variation from inner in the SMAT 10s Cu. Combined previous results (figure 2), the SMAT 10s Cu was further confirmed as BGSS. However, in figure 4(c), the SMAT 30s Cu exhibits a transitional region of grain size from surface to inner, which forms GS.
The kernel average misorientation (KAM) maps also verify BGSS and GS (figures 4(b), (d)). SMAT-ed Cu surface grains were plastic deformation, which cause strength difference between the hard domains and soft domains. GNDs are introduced into the interface between the hard domains and soft domains to maintain the structural continuity during tensile strain [28, 29].

3.2.2. TEM characterization
Dislocation slip and deformation twinning are the two deformation mechanisms of materials [30]. It was reported that the influence of stacking fault energy (SFE) on deformation mechanisms of materials have been extensively investigated [14, 31, 32]. As the region of 70 μm from the surface is less influenced by the deformation, the early structural evolution of the deformation can be observed. In figure 5(a), a large number of dislocations can be observed to be tangled. The dislocations appear in the shape of irregular waves and tend to form dislocation cells by tangling. Figure 5(b) shows the clear dislocation cell. The dislocation accumulation and forest hardening mechanisms can provide work hardening. The mechanisms of grain refinement can thus be determined.

In Cu with SFE (~45 mJ m⁻²), during the SMAT process, the surface of the sample was severely deformed by the continuous impact of the steel balls on the sample surface. A large number of dislocations were generated.

Figure 3. (a) Schematic diagram of the sampling location of the nano-indentation and hardness variation on the hard domain of the SMAT 5s Cu. The yellow line is the average hardness for the hard domain. (b) Vickers hardness variation combined with optical micrograph for the SMAT 30s Cu.

Figure 4. (a) Image quality (IQ) maps and (b) kernel average misorientation (KAM) maps of the SMAT 10s Cu. The insets are local zoom-in maps. (c) IQ map and (d) KAM map of the SMAT 30s Cu.
and accumulated inside the grains, followed by the formation of subgrain boundaries and grain boundaries, resulting in grain refinement, which is consistent with previous studies [30, 33].

### 3.3. Mechanical properties

Figure 6(a) shows the engineering stress-strain curves for the annealed and SMAT-ed Cu. The yield strength (YS) and uniform elongation (UE) of annealed Cu were about 53.8 MPa and 42.7%, respectively. After SMAT, the YS of the samples for different SMAT time (5 s, 10 s, 30 s, 60 s) were 71.5 MPa, 103.1 MPa, 146.7 MPa and 162.8 MPa, respectively. However, compared with the annealed Cu (UE of 42.7%), their UE decreased, corresponding to 40.8%, 34.7%, 29.4% and 27.4%, respectively. It can be seen that the SMAT 10s Cu had a high YS and reasonable ductility (figure 6(b)). For longer SMAT time Cu, such as the SMAT 60s Cu, YS was significantly improved but UE was reduced dramatically. Previous results [24] showed that the gradient structure volume fraction increased, and strength was improved but ductility was reduced with the increase of SMAT time. It indicates that the gradient structure volume fraction can tune the match of strength and ductility, which is agreement with previous study [29, 34, 35]. Figure 6(c) presents strain hardening rate ($\frac{\partial \sigma}{\partial \varepsilon}$) as a function of true strain. The SMAT-ed Cu has a lower strain hardening rate compared to the annealed Cu. However, the strain hardening rates of SMAT 5s and SMAT 10s Cu are higher than that of the SMAT 30s and SMAT 60s Cu, delaying the onset of necking. As a result, the SMAT 5s and SMAT 10s Cu exhibit better ductility.

According to previous reports [29, 34, 35], GNDs were introduced to accommodate mechanical incompatibility between hard domains and soft domains in heterogeneous structure, resulting in significant HDI strain hardening and stress strengthening. In present study, HDI effect were also observed both in SMAT 10s and SMAT 60s Cu (BGSS, GS, respectively), as shown in figure 7(a). The quantity of HDI stress in the figure 7(b) can be obtained from the previous studies [36, 37]. It can be found that HDI stress of SMAT 60s Cu was higher than that of SMAT 10s Cu under the same strain. Specifically, HDI stress of SMAT 10s Cu accounted for approximately 75% of that of SMAT 60s Cu. From figure 7(c), it is interesting to find that the HDI stress value of SMAT 10s Cu is approximately 75% of that of SMAT 60s Cu, almost similar to the hard domains (71.84%) on the SMAT 10s Cu surface (in figure 2).

It was reported that the magnitude of HDI stress is proportionally linked to the GNDs density [28, 38]. Based on HDI stress values (figure 7(b)) and surface morphology (figure 2), schematic diagrams of GNDs accumulation for the SMAT 10s and SMAT 60s Cu were illustrated in figures 7(d) and (e), respectively. In the SMAT 10s Cu with only partially refined grains (figure 7(d)), dislocations can be classified into two types, i.e., type I (GNDs) and type II (forest dislocations). Type I belongs to heterogeneous type with hard domains and soft domains and thus it can generate significant HDI effect. However, type II hardly produces the HDI effect owing to the homogeneous structures [39]. Surface grains of SMAT 60s Cu were sufficiently refined, the interface density between CG and refined grain was higher than that of SMAT 10s Cu with only partially refined grains. Therefore, more GNDs in the SMAT 60s Cu were formed to accommodate strain gradient at the interface, which was higher than that of SMAT 10s Cu. These results correspond to HDI stress values. Meanwhile, it indicates that completely hard domains on the SMAT surface can effectively accumulated GNDs [40].

HDI stress was found to increase with increasing SMAT time, while ductility of SMAT-ed Cu had a reduction to certain extent. The development of HDI stress is different from ductility in SMAT-ed Cu. With increasing SMAT time, the gradient structure volume fraction of SMAT-ed Cu was improved to result in enhancing strength and HDI stress. However, the gradient structure volume fraction was enhanced and corresponding volume fraction of CG was reduced, while CG can maintain ductility of SMAT-ed Cu. Therefore, optimized mechanical properties can be obtained by tuning the gradient structure volume fraction of SMAT-ed Cu.
3.4. In-situ DIC observation

The evolution of the strain distribution (along the y-direction) on the SMAT-ed surface were observed by DIC records and subsequent GOM software analysis with the increasing applied tensile strain, as shown in figure 8(a). In figure 8(b), it can be seen that no shear bands (SBs) appear on the surface of the annealed Cu and strain is uniform when applied tensile strain ($\varepsilon_{\text{app}}$) is below 25%. However, significant SBs appeared with the angle of 40–50° between the SBs and the horizontal direction in all SMAT-ed Cu. The distribution of SBs was denser in some regions at the same $\varepsilon_{\text{app}}$ with increasing the SMAT time. It indicates that SBs carry more strain than surrounding regions. A single strong SB usually leads to a localization of early strain, resulting in limited ductility of NS materials [41]. In this study, it should be noted that there is no direct connection between the strain evolution on the surface and the pits from figure 8(a). Therefore, it can be regarded that the hard domains formed by SMAT hardly cause strain concentration.

In order to analyze the constraining effect of soft domains on hard domains, the SMAT 60s Cu was peeled from one side of SMAT surface until only the GS region remained (the thickness of GS was determined by hardness distribution), and this sample was defined as SMAT 60s-GS Cu. As shown in figures 8(d), (e), in contrast to the SMAT 60s Cu, the SMAT 60s-GS Cu shows strong localized strain zone (LSZ) after yielding. The zone in which $\varepsilon_y$ is over $\varepsilon_{\text{app}}$ is defined as the LSZ. In addition, the strain is rapidly concentrated in the LSZ until failure, which is attributed to the low strain hardening ability for the SMAT 60s-GS Cu. However, in the SMAT 60s Cu, SBs are more uniformly distributed on the surface at the early strain stage due to the interaction between the hard domains and soft domains (figure 8(d)). It can be seen that these SBs have a more significant accumulation of strain, however, none of SBs cause strong LSZ to necking. Therefore, it can be regarded that these SBs are stable and maintain uniform strain of the material [42].

To further explore the surface strain distribution with increasing strain. Five intercept lines are selected in the DIC observed area along the y-direction (as figure 8(a)). And the data of lines are statistically analyzed to obtain figures 8(f), (g). In figure 8(f), when $\varepsilon_{\text{app}}$ is 10%, the strain distributions of the surface of the annealed and the SMAT 5s Cu are more uniform, while the strain distributions of the surface for SMAT 10s and SMAT 60s Cu have significant fluctuations, especially in the SMAT 60s Cu. The ratio of the average strain in the LSZ ($\bar{\varepsilon}_{\text{LSZ}}$) to $\varepsilon_{\text{app}}$ can be used to represent the degree of strain localization.

From figure 8(g), there is no significant strain localization in the annealed Cu. However, the SMAT-ed Cu show strain localization at the early strain stage, and for the longer the SMAT time (10s and 60s), samples exhibit the higher degree of strain localization. The strain localization diminished with increasing strain. It was related

---

Figure 6. (a) Engineering stress-strain curves and tensile test results for annealed and SMAT-ed Cu. (b) Variation of YS, UTS and UE versus different SMAT time. (c) Strain hardening rate curves as a function of true strain.
with the soft domains resistance to strain instability and effectively restrains development of strain at the LSZ in the early stage, resulting in uniformly distributed strain over the surface. Therefore, the soft domains stabilize strain, which effectively delays the onset of necking and contributes to reasonable ductility of SMAT-ed Cu with high strength simultaneously.

As shown in figure 8(h), the strain maximums on the surface during tensile process can be obtained by counting DIC observed area. This used to demonstrate the constraining effect of the soft domains on the local strain in heterogeneous materials (BGSS and GS). Annealed Cu shows the most stable strain evolution among all samples. The SMAT-ed Cu show more significant increase in the maximum strain at the early stage of tensile process ($\varepsilon_{\text{app}}$ below 2%). This is due to the presence of the hard domains and the formation of SBs on the surface. The SMAT 5s Cu almost shows the same trend that increase in maximum strain with that of the annealed Cu. This is attributed to the fact that the SMAT 5s Cu has fewer the hard domains, which effectively stabilizes the development of the SBs. However, due to formation of more hard domains in the SMAT 10s and 60s Cu, the soft domains fail to act as a constraint on the SBs. With increasing of strain, the restraining effect of the soft domains on the SBs gradually decreases, and the LSZ increases until failure.

It demonstrates that the SBs can maintain stable evolution and are uniformly distributed over the surface, maintaining the ductility of the SMAT-ed Cu. In addition, due to the interaction between hard domains and soft domains in heterogeneous structured materials, strain gradient is introduced at interface of the two domains, result in producing HDI stress and synergistic strain hardening [43–45].
3.5. Surface roughness

The soft domains start to deform plastically while the hard domains are still deforming elastically, leading to a rapid increase of GNDs and HDI stress. Moreover, the multi-axial stress states activate more slip systems, which contribute to dislocations interaction and strain hardening. In this study, to observe strain evolution of the soft domains and hard domains by 3D cross-sectional topography, the SMAT 60s and SMAT 10s Cu were taken as examples, as shown in figure 9. In figure 9(a), (b), the variation in height for the SMAT 60s and SMAT 10s Cu before and after tensile experiment were observed. To obtain data in detail, the average of the five intercepted lines (along the X axis) was calculated to reveal the cross-sectional strain evolution in figure 9(c). It can be found that there is not obvious variation in height before and after tensile strain (2%) for the SMAT 10s Cu. However, the SMAT 60s Cu has...
Figure 8. (Continued.)

Figure 9. 3D cross-sectional topography at ε_{app} of 0% and ε_{app} of 2% for the (a) SMAT 60s and (b) SMAT 10s Cu, respectively. (c) Height variation for the SMAT 10s and SMAT 60s Cu, respectively. The insets are the local zoom-in maps.
a significant variation in height between the hard domains and soft domains before and after tensile (2%), which indicate that there was a constraint effect between the surface layer and interior layer. For the SMAT 10s Cu, due to low hard domains area (71.84%) on the two sides of SMAT surface, the interior layer (CG) had a limited constraint effect on the surface layer. However, the SMAT 60s Cu formed a high hard domains area (100%) on the two sides of SMAT surface, contributing a high constraint effect of the interior layer (CG) on the hard domains. These results are consistent with the previous reports [29, 47].

4. Conclusions

In this study, two different types of heterogeneous structured (BGSS and GS) were prepared by controlling the SMAT time. Effect of bimodal grain size and gradient structure on HDI stress and mechanical properties was investigated, and the following conclusions can be drawn:

1. Combining surface morphology, hardness and microstructure, partially hard domains on the surface formed BGSS (SMAT 5s and 10s Cu), while the surface grains of the SMAT 30s and 60s Cu were sufficiently refined to form GS.
2. Tensile results showed that the strength of the sample increased with increasing the SMAT time, while the ductility decreased. In other words, strength and ductility could be optimized by controlling the volume fraction of gradient structure.
3. SMAT 60s Cu with GS exhibited higher HDI stress than SMAT 10s Cu with BGSS. It is related with higher accumulation of GNDs in SMAT 60s Cu.
4. The interaction between the surface layer and interior layer in SMAT 60s Cu was more pronounced than that of SMAT 10s Cu in terms of 3D cross-sectional topography. Furthermore, DIC results revealed that soft domains can effectively stabilize SBs development and spread the strain uniformly on the surface. This process delays the necking and results in combination of high strength and reasonable ductility.

Acknowledgments

The authors wish to thank Prof. Yuntian Zhu for his insightful and constructive comments and suggestions, and thanks for help of Shimadzu management at Tianhe District, Guangzhou. The authors would like to acknowledge financial support by the National Natural Science Foundation of China (NSFC) under Grants No. 51861015, No. 51664033, No. 51911540072, and 2019 JSPS/NSFC Bilateral Joint Research Project. The authors were supported by Basic Research Project of Yunnan Science and Technology Program under Grant No.202001AU070081. This work was also supported by JSPS Grants-in-Aid for Scientific Research (KAKENHI) Grants No.20K15064 and No. JP18H05256.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Xinkun Zhu https://orcid.org/0000-0002-2153-6125

References

[1] Kumar K S, Van Swygenhoven H and Suresh S 2003 Mechanical behavior of nanocrystalline metals and alloys Acta Mater. 51 5743–74
[2] Koch C C 2007 Structural nanocrystalline materials: an overview J. Mater. Sci. 42 1403–14
[3] Kang J, Kim J G, Park H W and Kim H S 2016 Multiscale architectured materials with composition and grain size gradients manufactured using high-pressure torsion Sci. Rep. 6 26390
[4] Edalati K and Horita Z 2016 A review on high-pressure torsion (HPT) from 1935 to 1988 Materials Science and Engineering-A-Structural Materials Properties Microstructure and Processing 652 325–52
[5] Wang J, Xu L, Wu R, Feng J and Zhang M 2020 Enhanced electromagnetic interference shielding in a duplex-phase Mg–9Li–3Al–1Zn Alloy processed by accumulative roll bonding Acta Metallurgica Sinica (English Letters) 7 490–9
[6] Fattah-ahlosesmi A, Gashiti S, Mazaheri O, Keshavarz Y and , K M 2016 Microstructure, mechanical properties and electrochemical behavior of AA1050 processed by accumulative roll bonding (ARB) Journal of Alloys and Compounds: An Interdisciplinary Journal of Materials Science and Solid-state Chemistry and Physics 688 44–55
[7] Islamgaliev R K, Nikitina M A, Ganeev A V and Shidikov V D 2019 Strengthening mechanisms in ultrafine-grained ferritic/martensitic steel produced by equal channel angular pressing Materials Science and Engineering 744 163–70
Wang Y M, Chen M W, Zhou F H and Ma E 2002 High tensile ductility in a nanostructured metal Nature 419 912–5

Fang T H, Li W L, Tao N R and Lu K 2011 Revealing extraordinary intrinsic tensile plasticity in gradient nano-grained copper Science 331 1587–90

Wu X, Yang M, Yuan F, Wu G, Wu Y, Huang X and Zhu Y 2015 Heterogeneous lamella structure unites ultrafine-grain strength with coarse-grain ductility Nano 112 14501–5

Wang Q, Yang Y, Jiang H, Liu C T, Ruan H H and Lu J 2014 Superior Tensile Ductility in Bulk Metallic Glass with Gradient Amorphous Structure, Scientific Reports 4 4757

Lu K and Lu J 2004 Nanostructured surface layer on metallic materials induced by surface mechanical attrition treatment Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing 375 38–45

Yang X, Ma X, Moering J, Zhou H, Wang W, Gong Y, Tao J, Zhu Y and Zhu X 2015 Influence of gradient structure volume fraction on the mechanical properties of pure copper Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing 645 280–5

Liu X, Wu K, Wu G, Gao Y, Zhu L, Lyu Y and Lu J 2016 High strength and high ductility copper obtained by topologically controlled planar heterogeneous structures Scr. Mater. 124 103–7

Chan H L, Ruan H H, Chen A Y and Lu J 2010 Optimization of the strain rate to achieve exceptional mechanical properties of 304 stainless steel using high speed ultrasonic surface mechanical attrition treatment Acta Mater. 58 5086–96

Tao N R, Wang Z B, Tong W P, Sui M L, Lu J and Lu K 2002 An investigation of surface nanocrystallization mechanism in Fe induced by surface mechanical attrition treatment Acta Mater. 50 4603–16

Zhu Y and Wu X 2019 Perspective on hetero-deformation induced (HDI) hardening and back stress Mater. Res. Lett. 7 5939–8

Li J, Weng G J, Chen S and Wu X 2017 On strain hardening mechanism in gradient nanostructures Int. J. Plast. 88 89–107

Cao Y, Ni S, Liao X, Song M and Zhu Z 2018 Structural evolutions of metallic materials processed by severe plastic deformation Materials Science & Engineering B- Reports 133 1–59

Zhang Y, Yang C, Zhou D, Zhe Y, Meng L, Zhu X and Zhang D 2019 Effect of stacking fault energy on microstructural feature and back stress hardening in Cu-Al alloys subjected to surface mechanical attrition treatment Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing 740 235–42

Gong Y L, Wang Z B, Tong W P, Sui M L, Lu J and Lu K 2002 An investigation of surface nanocrystallization mechanism in Fe induced by surface mechanical attrition treatment Acta Mater. 58 5086–5096

Mishra A, Mettak B K, Gregori F and Meyers M A 2007 Microstructural evolution in copper subjected to severe plastic deformation Experiments and Analysis Acta Materialia 55 2563–4

Wu X, Jiang P, Chen L, Yuan F and Zhu Y T 2014 Extraordinary strain hardening by gradient structure PNAS 111 7197–201

Wu X and Zhu Y 2017 Heterogeneous materials: a new class of materials with unprecedented mechanical properties Materials Research Letters 5 527–32

Yang M X, Yuan F P, Xie Q G, Wang Y D, Ma E and Wu X 2016 Strain hardening in Fe-16Mn-10Al-0.86C-5Ni high specific strength steel Acta Mater. 109 213–22

Yang M, Pan Y, Yuan F, Zhu Y and Wu X 2016 Back stress strengthening and strain hardening in gradient structure Mater. Sci. Eng. A 642 31–8

Jennings A T, Gross C, Greer F, Aitken Z H, Lee S W, Weinberger C R and Greer J R 2012 Higher compressive strengths and the Bauschinger effect in conformally passivated copper nanopillars Acta Mater. 60 3444–55

Hughes D A, Hansen N and Bammann D J 2003 Geometrically necessary boundaries, incidental dislocation boundaries and geometrically necessary dislocations Scr. Mater. 48 147–53

Zhu Y et al 2021 Heterostructured materials: superior properties from hetero-zone interaction Materials Research Letters 9 1–31

Yuan F, Yan D, Sun J, Zhou L, Zhu Y and Wu X 2019 Ductility by shear band delocalization in the nano-layer of gradient structure Mater. Res. Lett. 7 12–17

Wang Y F, Huang C X, He Q, Guo F J, Wang M S, Song L Y and Zhu Y T 2019 Heterostructured induced dispersive shear bands in heterostructured Cu Scr. Mater. 170 76–80

Wang Y, Huang C, Li Y, Guo F, He Q, Wang M, Wu X, Scattergood R O and Zhu Y 2020 Dense dispersed shear bands in gradient-structured Ni Int. J. Plast. 124 186–98

Huang C X, Wang Y F, Ma X L, Yin S, Hoeppel H W, Goeken M, Wu X L, Gao H J and Zhu Y T 2018 Interface affected zone for optimal strength and ductility in heterogeneous laminate Mater. Today 21 713–9

Wu X L, Jiang P, Chen L, Zhang F F, Yuan F P and Zhu Y T 2014 Synergistic strengthening by gradient structure Mater. Res. Lett. 2 185–91

Li J, Chen S, Wu X and Soh A K 2015 A physical model revealing strong strain hardening in nano-grained metals induced by grain size gradient structure Materials Science and Engineering a-Structural Materials Microstructure and Processing 620 16–21