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Mechanical Properties of Nano- and Submicrocrystalline Iron Subjected to Severe Plastic Deformation by Friction

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By using nanoindentation technique relationship between microstructure and mechanical parameters such as nanohardness H_h, plasticity characteristic δ_A, and Young's modulus E were found to be dependent on the grain size of the α-Fe subjected to severe plastic deformation by friction (SPDF) with argon atmosphere. Unlike fcc-metals in which the decreasing of grain size to 20 nm results in hardness growth accompanied by decreasing the plasticity, it was found the reverse effect in bcc-Fe, i.e. decreasing the grain size from 50 to 20 nm caused the decrease of hardness and increase of plasticity. Moreover, the decrease of Young's modulus E with decreasing the grain size down to 20 nm was detected in nanoindentation experiments.

Keywords: Iron, Nanocrystalline Structure, Nanoindentation, Young`s Modulus, Nanohardness, Plasticity Characteristic δ_A.

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1. INTRODUCTION

Processing routes developed for grain refinement of metallic materials down to several hundreds or even tens nanometres are of great attention over the last decade. Nano-crystalline materials with a large number of grain boundaries and triple junctions exhibit interesting combination of physical and mechanical properties, making them of growing attention to researches employed in scientific and engineering applications. However, these properties are strongly dependent on the processing route employed for producing the nanocrystalline materials [1]. Compared to the others processing routes severe plastic deformation (SPD) technique offers the essential advantages for ultra-grain refining the structure of metallic materials, making them of 100 % dense and free of contaminations. Among the other properties mechanical parameters of nanocrystalline and submicrocrystalline materials are of great importance. These materials demonstrate specific features of mechanical behaviour [1-7]. However, the mechanical characteristic of metallic materials refined down to nano- and submicrometer scale, which were published in the literature, are of controversial nature and their explanation is under dispute.

Although the effect of the grain size on the mechanical properties of nanostructured and nanocrystalline fcc-metals has been much studied [1–5], the data on their mechanical characteristics and, especially, plasticity properties are few in number. Plasticity of nanostructured bcc-metals (Cr, Mo, W) determined in convention test by tension was found to be such small that its values are usually not given [2]. Indentation technique opens wide scope for determining the plasticity characteristics δ_H/δ_A to estimate plasticity of the materials, which behave in a brittle manner in convention tests [8, 9].

The present study aims to investigate the specific features of mechanical behaviour of iron subjected to severe plastic deformation (SPD) and occurred due to friction with argon gas atmosphere.

2. EXPERIMENTAL

2.1 Material and processing procedure

Cylindrical samples of α-Fe with purity 99.9 wt. % and grain size about 80 μm were subjected to SPD by friction (SPDF) with argon gas by using the set up described elsewhere [10]. Deformation induced structure of the α-Fe arisen at the surface of cylindrical samples was composed of four distinctive structural sections including those refined down to nanometre, submicrometre, and micrometre scales [10].

2.2 Mechanical Testing

Load-displacement experiments were performed in cross-sectional structure of the cylindrical sample by using Nano Indenter IIR tester equipped by Berkovich pyramid. Set of mechanical characteristics including Young's modulus, E, nanohardness, H_h, and plasticity characteristic δ_A was determined in nanoindentation experiments. Young's modulus, *E*, and nanohardness, H_h, were determined according to demands of International standard ISO 14577-1:2002 (E) based on the test method procedure originally proposed by Oliver and Pharr [11]. Plasticity characteristics δ_A that is physically close to plasticity characteristic δ_H [8, 12] was derived using loading/unloading data, as shown in Fig. 1.

Plasticity characteristic δ_A is determined from relation $\delta_A = A_p/A_t$ where $A_t = A_e + A_p$ defines total indentation work while A_e and A_p define elastic and plastic components, respectively.

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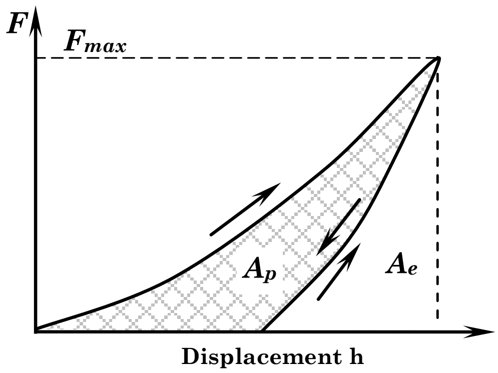

Fig. 1 – Schematic presentation of load-displacement diagram recorded in nanoindentation experiments

3. MECHANICAL PROPERTIES OF IRON SUBJECTED TO SPDF PROCESS

Fig. 2 shows mechanical characteristics via grain size of α-Fe subjected to SPDF with argon gas.

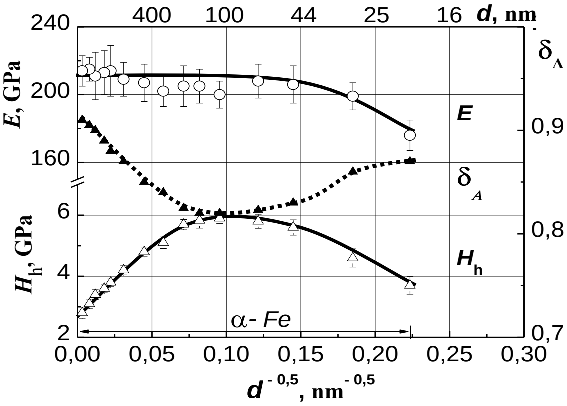

Fig. 2 – A summary of the data for mechanical characteristics such as nanohardness, H_h, plasticity characteristic, δ_A, Young's modulus, E, via grain size of the α-Fe subjected to SPDF

3.1 Young's Modulus

Young's modulus, E, for submicrocrystalline α-Fe remains the same as that for coarse grained matrix, as can be seen in Fig. 2. However, the decrease of grain size down to 30 nm causes Young's modulus to decrease by 10 % compared to that of coarse grained matrix.

Thus, SPDF of α-Fe with argon atmosphere leads to decrease in Young's modulus only at a grain size smaller than 30 nm. For larger grains the values of Young's modulus correspond to Young's modulus of coarse grained iron.

3.2 Nanohardness

After SPDF nanohardness, Hh, of α-Fe increases from 3 to 5.8 GPa as the grain size decreases from coarse grains about 80 μm to 200 nm, as shown in Fig. 2. For coarse grained α-Fe nanohardness, Hh, via grain size is satisfied to the Hall-Petch relation $H = H_0 + k_y d - 0.5$, where $k_y = 0.43$ MPa·m1/2, which almost coincides with this coefficient for ordinary polycrystalline α-Fe ($k_y = 0.39 - 0.73$) [13]. However, deviation of nanohardness from the Hall-Petch relation above arises when the grain size decreases from 200 to 50 nm, i.e. nanohardness, Hh, keeps constant values despite of decreasing the grain size. Furthermore, as evidenced from Fig. 2 nanohardness drops down sharply as grain size decreases roughly below 50 nm, implying the inverse of the usual Hall-Petch relationship.

3.3 Plasticity characteristic δ_A

Plasticity characteristic δ_A reflects nanohardness distribution via grain size, as can be seen in Fig. 2. Plasticity characteristic δ_A decreases with increasing the nanohardness values when grain size is ranged from 80 μm to 200 nm although it keeps constant values with further decreasing the grain size down to 50 nm. The important point concerns the fact that plasticity characteristic δ_A increases when the grain size decreases below 50 nm.

4. DISCUSSION OF RESULTS

The results of the present study show remarkable effect of ultrafine grain refinement on mechanical response of α-Fe. Generally, mechanical behaviour of metallic materials could be influenced by several structural parameters, i.e. the average grain size, dislocation density and other stacking faults as well as fraction volume of grain boundaries (GB) and triple junctions, and also grain boundary structure and grain boundary misorientation, i.e. either low or high angle GB. As applied to fine grained deformation structure induced by SPD, the precise contribution of each of the factors is yet open to question.

Young's modulus of α-Fe subjected to SPDF tends to be smaller with reducing the grain size down to 30 nm, as shown in Fig. 2. In literature several factors that affect the elastic properties of ultrafine-grained materials produced by SPD technique are discussed. Grain boundaries (GBs) and triple junctions are assumed to play a key role in the decrease of the elastic properties of metallic nanomaterials. This is because of significant fraction of atoms located at sites that differ from the usual positions in crystal lattice and the volume fraction of GBs and triple junctions increases as the grain size decreases [6, 14, 15]. According to the Mughrabi model for a composite materials [16, 17], Young's modulus E may decreases because of an increase of volume fraction of intergranular space filled by GBs and triple junctions, for which mechanical characteristics are rather different compared to those of the grain interior. Moreover, a decreasing the grain size leads to increasing the fraction of free volume generated by GBs, and triple junctions. As a result, the interatomic bonds in nanocrystalline material become weaker. That is why distorted elasticity of grain boundaries and triple junctions are considered to be responsible for decreasing the elasticity modulus of nanocrystalline material [2, 5, 6].

Nanohardness of ultrafine grained α-Fe resulted from SPDF is found to be in actual conflict with Hall-Petch relation (Fig. 2). According to the data published in [18, 19], deviation of nanohardness from the Hall-Petch relation is detected for nanocrystalline fcc-metals when grain size is reduced down to either 40 or 20 nm, i.e. the hardness decreases more slightly compared to that prescribed by the Hall-Petch relation [12, 18]. However, as evidenced from the data obtained in the present study, deviation from the Hall-Petch relation for the bcc-iron refined by SPDF is detected for larger grains, i.e. for grains refined to 200 nm and smaller.

According to modern concepts [18, 19], deviation of the nanohardness values from the Hall-Petch relation is resulted from the change in the deformation mechanism. Relay-race dislocation transfer mechanism indicative of metals with micrometre sized grain is gradually transformed to grain-boundary sliding and rotation with refining the grain size down to nanometre scale. It is noticeable that the change of deformation mechanism for bcc-iron is observed at the larger grains compared to that for fcc-metals. Coefficient k_y in the Hall-Petch relation for bcc-iron was believed to be close to those typical for the other bcc-metals [13] although it is much greater than those for fcc-metals, e.g. for copper [18]. This is the reason of increased sensitivity of deformation mechanism for bcc-iron to the nanograin size. Detailed explanation of the argumentation above was published in [12].

Plasticity characteristic δ_A is increases as the hardness decreases when Young's modulus keeps constant value [20]. The increase of plasticity characteristic δ_A becomes faster when Young's modulus decreases with decreasing the grain size roughly below 50 nm. It is noticeable that plasticity characteristic δ_A for bcc-iron with grain size of 20 nm achieves the value indicative of fcc-copper of the same nanograin size.

5. CONCLUSIONS

Remarkable effect of ultrafine grain refinement on mechanical response of bcc-metals was justified in nanoindentation experiments with α-Fe subjected to severe plastic deformation by friction (SPDF).

Set of mechanical characteristics including Young's modulus, E, nanohardness, H_h, and plasticity characteristic δ_A via grain size was determined and discussed.

When grain size decreases down to 30 nm Young's modulus decreases by roughly about 10 % compared to that indicative of coarse-grained α-Fe.

Nanohardness, H_h, of ultrafine grained α-Fe was found to be in actual conflict with the Hall-Petch relation. As-opposed to the Hall-Petch relation nanohardness, H_h, keeps constant values with decreasing the grain size from 200 to 50 nm. Furthermore, inverse of the Hall-Petch relationship was revealed when grain size of α-Fe decreases roughly below 50 nm.

Plasticity characteristic δ_A was found to increase as the hardness decreases when Young's modulus kept constant value although its increasing became faster when Young's modulus decreased with decreasing the grain size roughly below 50 nm.

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REFERENCES

1. *Nanostructured Materials: Scince and Technology*. (Dordrecht: Kluwer Acad. Publ., 1998).
2. R.A. Andrievski and A.M. Glezer, *Scripta Metall.* **44** N 8/9, 1624 (2001).
3. J.R. Weertman, *Mateer. Sci. Eng. A* **166**, 161 (1993).
4. K. Lu , M.I. Sui, *Scripta Met. Mater.* **28**, 1465 (1993).
5. D. Korn, A. Morsch, R. Birringer, and et al., *J. Phys. Coll. C5. Suppl.* **49** N 10, 769 (1988).
6. G. Palumbo, U. Erb, K.T. Aust, *Scripta Met. Mater.* **24**, 2347 (1990).
7. R.Z. Valiev, R.K. Islamgaliev, and I.V. Alexandrov, *Progr.Mater.Sci.* **45**, 103 (2000).
8. Yu.V. Milman, B.A. Galanov, S.I. Chugunova, *Acta Metall. Mater.* **41** N 9, 2523. (1993).
9. Yu.V. Milman, *J. Phys. D: Appl. Phys.* **41**, 1 (2008).
10. A.I. Yurkova, Yu.V. Milman, and A.V. Byakova, *Russian Metallurgy* (*Metally*) **2010** No4, 249 (2010).
11. W.C. Oliver and G.M. Pharr, *J. Mater. Res.* **7** No6, 1564 (1992).
12. A.I. Yurkova, Yu.V. Milman, and A.V. Byakova, *Deformaciya i Razrushenie Materialov* No**2**, 2 (2009).
13. V.I. Trefilov, V.F. Moiseev, E.P. Pechkovskii, et al., *Strain Hardening and Fracture of Polycrystalline Materials* (Kiev, Naukova Dumka: 1987).
14. R.A. Andrievskii and A.M. Glezer, *Phys. Met. Metallogr.* **88** No1, 45 (1999); *Phys. Met. Metallogr.* **89** No1, 83 (2000).
15. A.M. Glezer, in *Proceedings of the Ist International Conference on Deformation and Fracture of Materials,* (Moscow: 2006).
16. T. Ungar, H. Mughraby, D. Kцnnpagel, and M. Wilkens, *Acta Metall.* **32** No3, 333 (1984).
17. H. Mughraby, *Acta Metall.* **31**, 1367 (1983).
18. H. Van Swygenhoven and J.R. Weertman, *Materialstoday* **9** No5, 24 (2006).
19. K.S. Kumar, H. Van Swygenhoven, S. Suresh, *Acta Materialia* **51**, 5743 (2003).
20. Yu.V. Milman, B.A. Galanov, and S.T. Chugunova*, Acta Metal. Mater.* **41** No9, 2523 (1993).