On the atomic force microscopy characterization of void evolution in severely plastic deformed pure iron

N Forouzanmehr and N Nili-Ahmadabadi

School of Metallurgy and Materials engineering, University of Tehran, Iran
E-mail: nforouzanmehr@ut.ac.ir

Abstract. Different severe plastic deformation comprise equal channel angular pressing (ECAP), shaped cold rolling and drawing, or combined were applied on pure iron to obtain nano structured grains. The results show the formation of high concentration of excess free volume up to about 4% in the cold rolled and drawn specimens. Emphasis has been placed on atomic force microscopy (AFM) observations as additional characterization tools that complement the information provided by other techniques. Since the surface of the materials can be observed with atomic-scale resolution, the AFM is a powerful technique to study porous materials. The microscopy observations detect voids in the nanocrystalline Fe sample processed by shaped rolling followed by drawing with applied true strain of 7- from nano to sub-micrometer in size. It seems that the coalescence of nano-voids could lead to the formation of micro-voids in the structure of deformed samples.

1. Introduction

Severe plastic deformation (SPD) techniques can produce nanoscale and ultrafine-grain structures in a wide range of metals and alloys [1]. In order to impose an extremely large strain on the bulk metal, many SPD processes such as high-pressure torsion (HPT), equal channel angular pressing (ECAP), accumulative roll bonding (ARB), severe cold rolling, and deep wire drawing, etc have been developed [1-4].

It has been reported that excess free volumes or voids can form and grow at grain boundaries or at particle/matrix interfaces in materials at extremely large strains imposed by severe plastic deformation [5-8]. Non-equilibrium grain boundaries (GBs) of SPD-processed nanocrystalline metals possess excess energy, long-range stresses and enhanced free volume [9-11]. Free-volume type defects play a key role in the properties.

Study of structural features and void evolution in the course of severe plastic deformation is the subject of numerous investigations based on optical microscopy, scanning electron microscopy (SEM) and, for even higher resolution, transmission electron microscopy (TEM) studies [5-8]. Atomic force microscopy (AFM) as additional characterization tool that complement the information provided by these techniques has been more applied on materials science, but rarely used in voids study of SPD processed metals [12].

AFM is capable of quick and easy generation of 3D images of surface topography with nanometer resolution in which no vacuum is needed. This technique with scan sizes ranging from about 100 micrometer down to several nanometers is comparable to, in some cases, the high magnification achievable with TEM. Application of AFM to metal specimens allows detection of structural changes and defect on nanoscale and it is a well-established technique to study of porous materials [12].

The objective of this work is to study the microstructure of severely deformed pure Fe in various deformation processes, with combining the optical microscope, SEM and AFM observation.
2. Experimental

The initially material for severe deformation was commercially pure Fe. A sample with square cross section of $176 \, \text{mm}^2$ was deformed by shaped cold rolling to gain $\varepsilon \sim 4.6$, followed by wire drawing at ambient temperature to produce a wire with 0.45 mm in diameter and a large amount of strain of $\varepsilon \sim 7$. The other sample with rod shape and dimensions of $20 \times 20 \, \text{mm}^2$ at cross section and length of 60 mm are machined and prepared for ECAP deformation, through route A at room temperature and the constant frame velocity of 1 mm/s, up to 4 passes. The sections of the deformed specimens (in normal or rolling/drawing directions) were polished by standard metallographic procedures to a mirror like quality and were etched with 2% nital etchant. Microstructures of the samples were studied by a HITACHI field emission scanning electron microscope (FESEM) and atomic force microscopy (AFM) with a DualScope™ DS 95-200/50 AFM of Digital Instruments operated in tapping-mode and ambient air, using a silicon tip characterized by radius of curvature of less than 10 nm, an interior angle of less than 20° and a height of 15 µm. The density was determined using conventional Archimedes’ method to quantify the volume fraction of excess free volumes.

3. Results and Discussion

Fig. 1 shows the FESEM and AFM micrographs of shape cold rolled pure Fe with $\varepsilon \sim 4.6$ in rolling direction. Typical columnar grained microstructure elongated to the rolling direction with many shear bands which correspond to dislocation substructures was observed. With increasing strain, the initial grains were elongated more and more, and the spacing of grain boundaries decreased, so that the fine lamellar structure was achieved.

![Fig. 1 (a) FESEM and (b) AFM micrographs of shape cold rolled pure Fe with $\varepsilon \sim 4.6$ in rolling direction.](image-url)
The micrographs of the specimen deformed by shape rolling followed by drawing (SRD) with $\varepsilon \sim 7$ are represented in Fig. 2. As shown in Fig. 2, there are some voids from nano to sub-micrometer in size in the image. Such voids formation might be due to the formation of excess free volume in the material at high deformation strain. The voids are characterized by dark areas in the AFM image in Fig. 2(b), which confirm the FESEM observations. The depth of these voids was estimated from the cross sectional profiles to be up to about 300 nm. It should be noted that the AFM depth measurements are limited to the entrance of pores due to the differences between the geometry of the pore and the size of AFM tip probes for entrance to the pore. Therefore, the heights of these voids are likely to be more than the measurements. The coalescence of two micro-voids can be seen in cross sectional profiles of Fig. 2(b) as well as in Fig. 3 at longitudinal cross section of the specimen. Indeed by increasing deformation strain in wire drawing process, the formation of new voids and the growing and coalescence of the former voids have taken place. Mirsepasi et al. [13] presented the formation and coalescence of voids in the case of martensitic steel severely deformed by repetitive corrugation and straightening by rolling technique (RCSR), as shown in this study for pure iron processed by SRD, using FESEM and AFM. Although, the chemical etching can considerably change the size of the pore-type objects and optical microscopy, scanning electron microscopy and atomic force microscopy evaluate void formation with a certain level of uncertainty, it has been convinced that the void formation can be an inherent feature of the SPD processing and is not an artifact of etching [8].

![Fig. 2 (a) FESEM and (b) AFM micrographs of SRD processed Fe with $\varepsilon \sim 7$ in normal direction and related cross sectional profiles.](image)
Microstructure of Fe processed by ECAP after 4 passes was studied using optical microscopy as well as AFM. It was reported that various processing routes used in ECAP leads to different slip systems and structures. In route A, the specimen is pressed without rotation [14]. In this route, there is a cumulative build-up of additional strain on each separate pass through the die. Fig. 4 illustrates the cross sectional images of ECAP processed Fe in this study at normal direction. It can be seen that there are three distinguished areas which separate by nearly straight lines, as shown with dashed lines. There is a similarity between this structure and that from the implication of shearing systems which was reported for the route A in ECAP processing, using 90° die with rotation about the X axis as viewed in the four separate processing routes [15]. Thus, the middle area in the figure relates to shear banding. As can be seen it has very fine grained structure. AFM image of the sample in the central area that are demonstrated in Fig. 5 also represent shear banding. It is known that for large strains of pure metals the recrystallization temperature can be reduced below room temperature, resulted to recrystallization of the structure. Shear Bands (SBs) are suitable places to originate new grains under certain conditions during severe deformation. Optical microscopy can detect SBs by fine grains and provides direct information on shear band appearance [16].
Fig. 4 The cross sectional images of ECAP processed Fe in this study at normal direction.

Formation of voids similar to those were seen in the severely deformed Fe specimen processed by SRD, were not detected for the ECAP deformed Fe after 4 passes probably due to the mode of deformation which is applied by different SPD processes. However, the generation of microcrack after severe plastic deformation by ECAP was reported previously for Al-Mn alloy which are predominantly located in particles of the second phase and at interfaces of these particles [17].

Fig. 5 AFM images of the ECAP processed Fe at normal direction.

The observations were verified using density measurements. The variation of the bulk Fe density determined by the Archimedes' technique was shown in Fig. 6 for different SPD processes. For every sample, at least 8 readings were measured and the standard deviations were estimated. As can be seen, the density of shape rolled sample decreases. With increasing deformation strain, the formation of excess volume in the microstructure could be occurred, so that deformed Fe by SRD -with ε~7- has a high excess volume of ~4.03%.

On the other hand, the density of the ECAP deformed Fe compared with the shaped rolled Fe (with approximately equivalent strain) is almost the same as as-received iron. ECAP processing imposes hydrostatic pressure and can even reduce the total volume fraction of voids in the materials [18].
Fig. 6 Bulk Fe density in different severe deformation processes.

Although the generation of high excess free volume in pure Fe during SPD process has been previously reported [19], the formation of such a high concentration of free volume as voids—about 4%—has not been observed. Such voids may be formed by a number of mechanisms as reported in the literatures [5-7,20]. High total area of grain boundaries in nanocrystalline severely deformed materials increases the availability of void nucleation sites. Triple junction disclination, which represent typical junction defects in nanocrystalline materials, plays an important role in micro-crack generation processes at grain boundaries [5]. Theoretical analysis also suggests that micro-voids may form in metals under SPD, if the local maximum shear stress exceeds the shear yield stress [6]. Furthermore, during SPD processing at ambient temperature very high concentrations of vacancy type defects and vacancy agglomerates have been found that may promote cavitation [7]. Adiabatic shear bands also sometimes act as sites for voids nucleation [20].

4. Conclusions

In the present study, the microstructure severely deformed Fe has been investigated using FESEM and AFM. The observations show the shear banding in the specimens in different deformation processes. Density measurements show a sharp decline for severely deformed Fe by a combination of cold rolling and wire drawing, compared with undeformed Fe. The density measurement confirms the microscopy studies for presence of voids in the microstructure. The existence of voids and the coalescence of them were also shown clearly by AFM.
References

[1] R.Z. Valiev, R.K. Islamgaliev and I.V. Alexandrov 2000 Prog. Mater. Sci. 45 103.
[2] K. Edalati, T. Fujioka and Z. Horita 2009 Mater. trans. 50 44.
[3] H. Ghasemi-Nanesa, M. Nili-Ahmadabadi, A. Mirsepasi and C. Zamani 2013 Met. Mater. Int. article in press.
[4] K. Hanazaki, N. Shigeiri and N. Tsuji 2010 Mater. Sci. Eng. A 527 5699.
[5] K. Zhou, M.S. Wu and A.A. Nazarov 2008 Acta Mater. 56 5828.
[6] S.V. Divinski, K.A. Padmanabhan and G. Wilde 2011 Philos. Mag. 1.
[7] D. Setman, E. Schafer, E. Korznikova and M. J. Zehetbauer 2008 Mater. Sci. Eng. A 493 116.
[8] R. Lapovok, D. Tomus, J. Mang, Y. Estrin and T.C. Lowe 2009 Acta Mater. 57 2909.
[9] I. Sabirov, M.Yu. Murashkin and R.Z. Valiev 2013 Mater. Sci. Eng. A 560 1.
[10] A.A. Nazarov, A.E. Romanov and R.Z. Valiev 1995 NanoStruct. Mater. 6 775.
[11] R. Würschum, B. Oberdorfer, E-M. Steyskal, W. Sprengel, W. Puff, P. Pikart, C. Hugenschmidt and R. Pippan 2012 Phys. B 407 2670.
[12] J.I. Paredes, A. Martínez-Alonso and J.M.D. Tascón 2003 Microporous and Mesoporous Mater. 65 93.
[13] A. Mirsepasi, M. Nili-Ahmadabadi, M. Habibi-Parsa, H. Ghasemi-Nanesa and A. F. Dizaji 2012 Mater. Sci. Eng. A 551 32.
[14] R.Z. Valiev and T.G. Langdon 2006 Progress in Materials Science 51 881.
[15] M. Furukawa, Z. Horita and T.G. Langdon 2002 Materials Science and Engineering A 332 97.
[16] V.M. Segal 2002 Materials Science and Engineering A 338 331.
[17] V. I. Betekhtin, V. Skleniacka, I. Saxl, B. K. Kardashev, A. G. Kadomtsev, and M. V. Narykova 2010 Physics of the Solid State 52 8 1629.
[18] R. Lapovok, D. Tomus, J. Mang, Y. Estrin and T.C. Lowe 2009 Acta Mater. 57 2909.
[19] B. Oberdorfer, B. Lorenzoni, K. Unger, W. Sprengel, M. Zehetbauer, R. Pippan and R. Würschum 2010 Scr. Mater. 63 452.
[20] T.W. Wright 2002 The Physics and Mathematics of Adiabatic Shearbands First published Cambridge University.