Formation of nanocrystalline surface layers in various metallic materials by near surface severe plastic deformation

Masahide Sato\textsuperscript{a,*}, Nobuhiro Tsuji\textsuperscript{b}, Yoritoshi Minamino\textsuperscript{b}, Yuichiro Koizumi\textsuperscript{b}

\textsuperscript{a}Graduate School of Engineering, Osaka University, 2-1 Yamadaoka, Suita, Osaka 565-0871, Japan
\textsuperscript{b}Department of Adaptive Machine Systems, Graduate School of Engineering, Osaka University, 2-1 Yamadaoka, Suita, Osaka 565-0871, Japan

Received 29 August 2003; revised 29 September 2003; accepted 8 October 2003

Abstract

The surface of various kinds of metallic materials sheets were severely deformed by wire-brushing at ambient temperature to achieve nanocrystalline surface layer. The surface layers of the metallic materials developed by the near surface severe plastic deformation (NS-SPD) were characterized by means of TEM. Nearly equiaxed nanocrystals with grain sizes ranging from 30 to 200 nm were observed in the near surface regions of all the severely scratched metallic materials, which are Ti-added ultra-low carbon interstitial free steel, austenitic stainless steel (SUS304), 99.99 wt.% Al, commercial purity aluminum (A1050 and A1100), Al-Mg alloy (A5083), Al-4 wt.% Cu alloy, OFHC-Cu (C1020), Cu-Zn alloy (C2600) and Pb-1.5% Sn alloy. In case of the 1050-H24 aluminum, the depth of the surface nanocrystalline layer was about 15 \textmu m. It was clarified that wire-brushing is an effective way of NS-SPD, and surface nanocrystallization can be easily achieved in most of metallic materials.

© 2003 Elsevier Ltd. All rights reserved.

Keywords: Ultra-grain refinement; Nanocrystalline materials; Surface modification; Severe plastic deformation; Grain size; Microstructure; Steel; Aluminum alloy; Copper alloy; Lead alloy

1. Introduction

Recently, ultra-grain refinement or nanocrystallization of bulk structural materials has been energetically studied, because the ultrafine grained or nanocrystalline materials are expected to bear superior mechanical properties to conventionally coarse grained materials. The severe plastic deformation (SPD) processes, such as Accumulative Roll-Bonding (ARB) \cite{1–3}, Equal Channel Angular Pressing (ECAP) \cite{4,5}, High Pressure Torsion (HPT) \cite{6}, have been established, and they succeeded in forming the ultrafine grains (UFG) of mean grain sizes smaller than 1 \textmu m in various kinds of metallic materials \cite{3}. These UFG materials perform the strength two to three times higher than that of the starting ones \cite{1,2,7–10}, while the uniform elongation decreases with increasing the strength in the many UFG materials \cite{10}. In addition, all the SPD processes to produce bulk UFG materials need large amount of plastic-working energy as well as special equipments and/or techniques \cite{3}. By the way, the ultra-grain refinement only in near surface layers might be accomplished by simpler processes with less amount of plastic-working energy than the ultra-grain refinement of bulk materials. The materials having the UFG or nanocrystalline surface layers are expected to perform new superior properties, such as inhibition of fatigue crack initiation, high corrosion resistance, and applicability to functional coatings, because the surface is closely related with various properties of the materials.

In order to obtain the UFG or nanocrystalline structure in surface layers, near surface-severe plastic deformation (NS-SPD) process was applied to the surface of various metallic materials in the present study. Simple wire-brushing was used as the NS-SPD process. The wire-brushing induces intense strain to the surface layers of the materials. Surface nanocrystallization processes have been reported in several literatures \cite{11–17}. The nanocrystalline structure were, in most cases, accidentally formed by conventional processes such as hot-rolling \cite{11,12} and friction test \cite{13,14}, but some were intentionally developed by the process similar to shot peening \cite{15–17}. However, the number of such reports...
is still limited. The present study is a trial of simple NS-SPD easily applicable to practical use. The aim of the present study is to form surface nanocrystalline layers on various kinds of metallic materials by the NS-SPD using wire-brushing. The obtained surface layers are characterized by transmission electron microscopy (TEM) and the formation mechanism of nanocrystals is discussed.

2. Experimental

Ti-added ultra-low carbon interstitial free steel (IF steel, cold-rolled and fully annealed), austenitic stainless steel (JIS-SUS304, fully annealed), 99.9% aluminum (97% cold-rolled), 99.5% aluminum (JIS-A1050-H24), 99% aluminum (JIS-A1100-O), Al–Mg alloy (JIS-A5083-O; Al-4.5%Mg), Al-4 wt%Cu (96% cold rolled), OFHC-Cu (JIS-C1020-1/2H), Cu–Zn alloy (JIS-C2600-1/2H) and Pb-1.5%Sn alloy (89% cold-rolled) were provided to the NS-SPD in the present study. The chemical compositions of the materials except for that of the commercial SUS304 steel are listed in Table 1. The Al-4%Cu alloy was made by melting the 99.99%Al shown in Table 1 and 99.9% electrodeposited Cu and then rolling. The dimensions of the starting sample sheets were 1.0–2.0 mm in thickness, 20–40 mm in width and 100–200 mm in length. The grain sizes of the prepared metal and alloy sheets were 18, 15, 17, 15, 10, 17, 4 and 6 μm for IF steel, SUS304 steel, 99.99%Al, 1100Al–O, 5083Al–O, OFHC-Cu, Cu–Zn and Pb-1.5%Sn alloy, respectively. The 1050Al-H24 and Al-4%Cu sheets showed conventionally elongated deformation structures. After degreasing by acetone, the surface of the materials were wire-brushed at ambient temperature for the NS-SPD. A 304 stainless-wire bevel brush was driven by a hand grinder at a rotating speed of 12000 rpm. The number of the wires in the bevel brush was 2500, and the diameter and length of each wire were 0.3 mm and 14 mm, respectively. The rotating wire-brush was pressed on the surface of the sheets, and it was moved in both directions along the longitudinal direction (LD) of the sheets. The wire-brush rotated clockwise when observed from the driven side and it was tilted 15 degrees during moving, so that only the outer circumferential part of the wires hit the sample. The load applied to the sheets during wire-brushing was 3–5 kgf for steels and copper alloys, 1–3 kgf for aluminum alloys, and 0.5 kgf for Pb alloy. The moving speed of the wire-brush along LD was 18–35 mm/s for all the materials. The procedure described above was repeated up to about five times.

The microstructural observations from the normal direction (ND) of all the surface severely deformed sheets were conducted by TEM. Thin disc samples 3 mm in diameter including the severely deformed surface were cut from the sheets by using an electro-spark discharge machine. These discs were mechanically polished down to a thickness of 40–50 μm for the steels and the lead alloy, and 90–100 μm for the aluminum alloys and the copper alloys using 600–1500 grind papers only from the opposite side of the severely deformed surface. Then, the thin foils were prepared by twin-jet electropolishing after painting lacquer on the severely deformed surface to maintain the original surface. In electropolishing, a 10% HClO4 + 90% CH3COOH solution was used at 283 K for the steels, a 33% HNO3 + 67% CH3OH solution at 253 K for the aluminum alloys, a 30% HNO3 + 70% CH3OH solution at 258 K for the OFHC-Cu and the Cu–Zn alloy, and a 30% HClO4 + 70% CH3COOH solution at 283 K for the lead alloy, respectively. In addition, the surface layer of the 1050-H24 aluminum sheet was observed also from the transverse direction (TD) of the sheet. The thin foil perpendicular to TD was prepared by means of ultramicrotomy. Hitachi H-800 electron microscope was operated at 200 kV for the TEM observations. The mean grain sizes of the microstructures were measured from the TEM micrographs.

Table 1
Chemical compositions of the materials studied (mass%)

|      | C   | N   | O2  | Mg  | Al  | Si  | P   | S   | Ca  | Ti  | Cr  | Mn  | Fe  | Ni  | Cu  | Zn  | Sn  | Sb  | Pb  |
|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| IF   | 0.002 | 0.003 |     |     |     |     | 0.01 | 0.012 | 0.072 | 0.17 | bal. | 0.02 | 0.01 |
| 99.9%Al |     |     |     | 99.993 |     | 0.03 |     |     |     |     |     |     |     |     |     |     |     |     |     |
| 99.5%Al (A1050) | 0.00 | 99.59 | 0.08 |     |     |     | 0.03 | - | 0.00 | 0.27 | 0.01 | 0.001 |
| 99%Al (A1100) | 0.02 | 99.14 | 0.11 |     |     |     | 0.03 | 0.00 | 0.01 | 0.56 | 0.12 | 0.01 |
| Al-4.5%Mg (A5083) | 4.42 | bal. | 0.08 |     |     |     | 0.01 | 0.09 | 0.62 | 0.25 | 0.03 | 0.01 |
| OFHC-Cu (C1020) | 0.0002 | 0.0001 | 0.0003 |     |     |     |     |     |     |     | 0.0002 | bal. |     | 0.0001 |     |     |     |     |
| Cu–Zn (C2600) |     |     |     |     |     |     |     |     |     |     |     |     |     | 0.0043 | 69.38 | bal. | 0.0022 |
| Pb-1.5%Sn | 0.06 |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     | 1.5 | bal. |
3. Results

TEM microstructures and corresponding selected area diffraction (SAD) patterns of the surface in all the NS-SPD metallic materials are shown in Fig. 1. The microstructures were observed from the ND. The SAD patterns in Fig. 1 were taken by the use of an aperture with a diameter of 1.6 μm except for the case of 99.99% Al using a 4.4 μm aperture. In all the severely deformed materials, almost equiaxed nanocrystals were observed. However, the nanocrystalline microstructures were not very distinct either in the materials having the high melting point, such as IF steel and SUS304 steel or in the materials including relatively large amount of alloying elements, such as 5083-O aluminum, Al-4%Cu and brass. All the SAD patterns show ring-like patterns, which suggests that the nanocrystals are polycrystals with large misorientations to each other.

Fig. 2 shows the histograms of the grain sizes in the surface layers. The grain sizes are distributed from about 30 to about 400 nm. The alloys such as SUS304, 5083Al–O, Al-4%Cu and Cu–Zn have the grain sizes with narrow distribution compared with pure metals such as 99.99% Al and OFHC-Cu. When five kinds of aluminum alloys are
compared to each other, the grain sizes of the nanocrystals tend to increase with increasing the purity of the alloys. The similar trend that the grain sizes of metals are larger than those of alloys is recognized between IF steel and SUS304 and between OFHC copper and brass. It is noteworthy that brass, which is known to have very low stacking fault energy, showed the smallest grain size (29 nm).

Fig. 3 shows the TEM microstructure on the longitudinal section including the severely deformed surface of the 1050-H24 aluminum. The corresponding SAD patterns at various depth positions are also shown in the figure. A dark band-like patterns parallel to the sheet surface around the 20 μm depth position in Fig. 3 are a kind of damage formed during sectioning by ultramicrotomy. The microstructure of the area from the surface to about 10 μm depth position is evidently different from that at deeper depth position. The UFGs with grain sizes much smaller than 1 μm were observed in the subsurface region. On the other hand, the coarse grains or subgrains were observed in the internal regions below 20 μm, although the grains were not distinct in these inside regions due to the dislocation substructures or the patterns formed during thin foil preparation.

While the SAD patterns of the surface layer with the nanocrystalline structure show ring-like patterns (see A, B and C Fig. 3), the SAD patterns of the inside regions are spotty ones (D–F Fig. 3), which corresponds well with the transition in the microstructure described above. The high-magnification TEM microstructures at two different depth positions are shown in Fig. 4. The nanocrystals whose grain sizes are smaller than 100 nm are observed at the depth position of about 9 μm from the surface (Fig. 4(a), surface layer). The nanocrystals were elongated slightly along ND. The grain sizes \( d_{LD} \) and \( d_{ND} \) in parallel directions to LD and ND are 47 nm and 69 nm, respectively. On the other hand, the microstructure at the deeper depth position of about 34 μm (Fig. 4(b), internal region) is obviously different from that in the surface layer, and it appears to be conventionally deformed microstructure. This indicates that the nano-crystalline microstructures with large misorientations, i.e. polycrystalline nano-grains, were successfully introduced at the surface layers by the present NS-SPD process. Fig. 5 shows the grain/subgrain size \( d \) in the 1050-H24 aluminum as a function of the depth position from the surface. The grain sizes \( d_{LD} \) and \( d_{ND} \) in parallel directions to LD and ND, and the averages \( d_{AVE} \) of \( d_{LD} \) and \( d_{ND} \) were...
The nanocrystals of approximately 100 nm grain size exist in the area from the surface to the depth position of about 17 μm. The grain size increases slightly with increasing the depth. On the other hand, the grain/subgrain size suddenly increases at the depth position of about 18 μm, corresponding to the obvious transition in the microstructure as was shown in Fig. 3. The nano-grains were elongated slightly along ND in all the depth position. This probably reflects the metal flow in the NS-SPD by wire-brushing, though the details of it is unclear. From this variation in the grain size along with the depth, it is confirmed that the wire-brushing can severely deform the near surface layer of 1050-H24 aluminum up to the thickness of 17 μm to realize nanocrystalline structure.

4. Discussion

The formation process of the UFGs in bulk materials during severe plastic deformation had been investigated, and the following mechanism was proposed [18,19]. Many kinds of deformation boundaries are introduced within the original grains during plastic deformation. They are classified into two types of boundaries: incidental
dislocation boundary (IDB) and geometrically necessary boundary (GNB) [18,19]. The GNBs are formed due to difference in slip patterns, i.e. the combination and/or amount of the operated slip systems, between adjacent regions, and the GNBs usually have large misorientation after relatively large plastic strain. The process during which those boundaries are introduced is called grain subdivision [18,19]. In case of the severe plastic deformation above equivalent strain of 4, original crystals are finely subdivided in submicrometer scale by many GNBs. This ultrafine grain subdivision is the essentially important process for the ultrafine or nano grains formation during SPD [20,21]. However, the GNBs in the as-deformed state should be composed of (or decorated by) dislocations. Therefore, recovery to rearrange the dislocations at the boundaries is necessary to form obvious grain boundary, in other words, obvious UFG. In case of the materials relatively easy to recover, the rearrangement process can occur during processing simultaneously due to heat generation, so that clear ultrafine or nano grains can be observed even in the as-deformed state [20,21]. On the other hand, the complicated and unclear structures are often observed in the as-deformed materials having relatively high melting point (such as steels) because of the difficulty in recovery at low temperatures. Even in these cases, however, clear UFG form by recovery during subsequent annealing at relatively low temperatures [20,21]. Anyhow, the formation process of the UFG during SPD can be understood as a combination of the ultrafine grain subdivision and recovery. The present nanocrystal formation in the surface layers by near surface-severe plastic deformation (NS-SPD) processing should be explained in terms of the ultrafine grain subdivision and recovery. The wire-brushing severely scratches and stirs the metals in the surface layers, so that complicated metal flow is induced there. Such complicated metal flow gives rise to very large amount of plastic strain into the surface layer, which realizes nano-scale grain subdivision. The SAD patterns observed at the surface layers showed ring-like shape indicating large misorientations in small regions. This suggests that very high density of GNBs were introduced in the surface layers by the present NS-SPD. Because of the very high strain at high speed, fairly large increase in temperature occurred during the processing, which would assist the recovery. As a result, clear nanocrystals were observed in the materials relatively easy to recover, such as pure aluminum, in Fig. 1. In case of highly pure aluminum, further, grain growth could occur subsequently, resulting in larger grain sizes (also in Fig. 1). On the other hand, very fine but unclear structures were observed in the materials having high melting temperature, such as steels. Even for these materials, however, clear nanocrystals should form after annealing, if the formation mechanism is the same as that described before. Fig. 6 shows the microstructures of the IF steel NS-SPD processed at room temperature and then annealed at 500 °C for 600 s. Compared with
the as-processed microstructure in Fig. 1(a), clear nano-grains are observed. This coincides with the previously reported fact in the severely deformed bulk low-carbon steel which showed only complicated deformation structure but clear UFG after warm-temperature annealing [20,21]. Thus, the formation process of the nanocrystals in the surface layers in the present study is thought to be the same as that in the SPD of bulk materials, i.e. nano-scale grain subdivision and subsequent recovery.

In most cases, the minimum grain sizes induced by the ultragrain refinement of bulk SPD are more than 100 nm, independent of the process. On the other hand, the grain sizes of the surface UFG obtained in the present study are several tens nm. This is presumably because of the larger strain and higher strain rate during the NS-SPD than those in bulk SPD, although it is difficult to evaluate these values of strain and strain rate quantitatively at the present moment. Further, the increase in temperature is also important. Tsuji et al. [22] recently clarified that the ultrafine grain size in a commercial purity aluminum (1100) approaches a constant value with increasing strain above four during bulk SPD by the ARB process, because of the grain growth enhanced by heat generation during large plastic deformation. The surface layer in the present investigation is also heated up by the NS-SPD, but the surface layers are rapidly cooled just after the NS-SPD because the volume of the deformed region is small in the whole materials and the heat will diffuse into the inner part and the atmosphere. It is considered therefore that the grain growth hardly occurs in the NS-SPD, which maintains nanocrystals even in high-purity aluminum. This is a great advantage of the NS-SPD compared with the bulk SPD.

The UFG at near surface regions have been also reported by other researchers. Scamans et al. [11] and Leth-Olsen et al. [12] reported that the hot rolled aluminum alloys have the surface layer having nano-grains whose grain sizes are several tens nano meters. This nanocrystalline microstructure was formed presumably by a kind of SPD due to large friction between the roll and the specimen during hot-rolling. However, the thickness of the surface nano layer was very small, 1 µm. Hughes et al. [13,14] found that the surface layers of pure copper deformed under the friction test with large sliding loads have the ultra fine lamellar boundary structures which were similar to those observed in heavily rolled materials [20], and that the lamellar boundary structures has the average boundary spacing ranging from 50 to 170 nm at the depth positions from 2 to 20 µm, respectively. The local deformation occurs in the subsurface zones at a sliding interface during the friction test, and the UFG structure is formed. Tao et al. [15,16] clarified that the nanostructured surface layer whose grain sizes were about 10 nm at the top surface, about 100 nm at the 15 µm depth position and about 1 µm at the 40 µm depth position was formed in a pure Fe plate by means of surface mechanical attrition treatment (SMA), which is similar to shot-peening. Unemoto et al. [17] also reported that the nanocrystalline structure on the surface of steels was formed by air blast shot peening, although the detail profiles of grain size had not been shown. In those previous reports, the plastic deformation in the surface layers leads to the UFG structures. In the deformation by the hot rolling [11,12] and sliding [13,14] as method above, the plastic deformations are imposed to the surface layers by unidirectional shear strain. These processes give rise to the maximum strain at the top surface, and the strain decreases gradually with increasing the distance from the surface. In SMA [15,16] and air blast shot peening [17] where the vertical impact loads to the surface are applied, the maximum plastic strain is thought to be produced at the surface. In this case, the strain gradually decreases as the depth increases again, and similarly, the grain size gradually increases as that increases. On the other hand, the plastic deformation given by the present NS-SPD seems constant within the nanocrystalline surface layers, because the grain size in Fig. 5 was nearly constant. This can be also another
advantageous point of the present NS-SPD to obtain uniform nanocrystalline surface. Furthermore, the thickness of the present surface nanocrystalline layers (about 15 μm) in aluminum is much larger than those in aluminum alloys reported by Scamans et al. (1 μm) [11] and Leth-Olsen et al. (1 μm) [12] and comparable to those in copper or iron by Hughes et al. (10 μm) [13], Tao et al. (15 μm) [15] and Umemoto et al. (5 μm) [17]. Because several parameters, such as load, rotating speed and proceeding speed, in the present process are changeable, it would be possible to control the thickness of the nanocrystalline layers which must affect the properties of the materials. It should be emphasized, anyway, that the present NS-SPD is the simplest and most promising process for producing surface nanocrystalline materials at this moment.

5. Conclusions

The surface nanocrystalline layers were successfully formed in various kinds of metallic materials by means of the NS-SPD process using wire-brushing. The grain sizes in the NS-SPD processed surface layers were 30–200 nm. In case of the 1050-H24 aluminum, the surface nanocrystalline layers had thickness of about 15 μm. The formation of the nanocrystalline surface layers is explained by nano-scale grain subdivision and recovery during the NS-SPD. The present NS-SPD process is effective and useful to form nanocrystalline structure in the surface layers of materials. It could be concluded that a new and simple process to obtain surface nanocrystalline metallic materials was developed.

Acknowledgements

The present study was financially supported by the 21st century COE program (the Center of Excellence for Advanced Structural and Functional Materials Design in Osaka University) through the Ministry of Education, Sports, Culture, Science and Technology of Japan, and by Industrial Technology Research Grant Program ‘01 from NEDO of Japan under project ID 01A23025d. The supports are gratefully appreciated by the authors.

References

[1] N. Tsuji, Ultrafine grained steels, Tetsu-to-Hagane 88 (2002) 359–369.
[2] Y. Saito, H. Usunomiya, N. Tsuji, T. Sakai, Novel ultra-high straining process for bulk materials—development of the accumulative roll-bonding (ARB), Acta Mater. 47 (1999) 579–583.
[3] N. Tsuji, Y. Saito, S.H. Lee, Y. Minamino, ARB (Accumulative Roll-Bonding) and other new techniques to produce bulk ultrafine grained materials, Adv. Eng. Mater. 5 (2003) 338–344.
[4] R.Z. Valiev, E.V. Kozlov, Y.U.F. Ivanov, J. Lian, A.A. Nazalov, B. Baudelet, Deformation behaviour of ultra-fine-grained copper, Acta Metall. 42 (1994) 2467–2475.
[5] H. Hasagawa, S. Komura, A. Usunomiya, Z. Horita, M. Furukawa, M. Nemoto, T.G. Langdon, Thermal stability of ultrafine-grained aluminum in the presence of Mg and Zr additions, Mater. Sci. Eng. A 265 (1999) 188–196.
[6] R.Z. Valiev, A.V. Kornikov, R.R. Mulyukov, Structure and properties of ultrafine-grained materials produced by severe plastic deformation, Mater. Sci. Eng. A 168 (1993) 141–148.
[7] Y. Saito, N. Tsuji, H. Usunomiya, T. Sakai, R.G. Hong, Ultra-fine grained bulk aluminum produced by accumulative roll-bonding (ARB) process, Scripta Mater. 39 (1998) 1221–1227.
[8] N. Tsuji, Y. Saito, H. Usunomiya, T. Yamane, Ultra-fine-grained bulk steel produced by accumulative roll-bonding (ARB) process, Scripta Mater. 40 (1999) 795–800.
[9] Y. Ito, N. Tsuji, Y. Saito, H. Usunomiya, T. Sakai, Change in microstructure and mechanical properties of ultra-fine grained aluminum during annealing, J. Jpn Inst. Met. 64 (2000) 429–437.
[10] N. Tsuji, Y. Ito, Y. Saito, Y. Minamino, Strength and ductility of ultrafine grained aluminum and iron produced by ARB and annealing, Scripta Mater. 47 (2002) 893–899.
[11] G.M. Scamans, A. Afseth, G.E. Thompson, X. Zhou, Ultra-fine grain sized mechanically alloyed surface layers on aluminum alloys, Mater. Sci. Forum. 396-402 (2002) 1461–1466.
[12] H. leth-Olsen, J-H. Nordlien, K. Nisacioglu, Filiform corrosion of aluminum sheet III. Microstructure of reactive surface, Corros. Sci. 40 (1998) 2051–2063.
[13] D.A. Hughes, N. Hansen, Graded nanostructures produced by sliding and exhibiting universal behavior, Phys. Rev. Lett. 87 (2001) 135503/1–135503/4.
[14] D.A. Hughes, D.B. Dawson, J.S. Korellis, L.L. Weingarten, Near surface microstructures developing under large sliding loads, J. Mater. Eng. Per. 3 (1994) 459–475.
[15] N.R. Tao, Z.B. Wang, W.P. Tong, M.L. Sui, J. Lu, K. Lu, An investigation of surface nanocrystallization mechanism in Fe induced by surface mechanical attrition treatment, Acta Mater. 50 (2002) 4603–4616.
[16] N.R. Tao, M.L. Sui, J. Lu, K. Lu, Surface nanocrystallization of iron induced by ultrasonic shot peening, NanoStruct. Mater. 11 (1999) 433–440.
[17] M. Umemoto, Y. Todaka, K. Tsuchiya, Formation of nanocrystalline structure in steels by air blast shot peening, Mater. Trans. 44 (2003) 1488–1493.
[18] N. Hansen, D.J. Jensen, Development of microstructure in FCC metals during cold work, Phil. Trans. R. Soc. Lond. A 357 (1999) 1447–1469.
[19] D.A. Hughes, N. Hansen, High angle boundaries formed by grain subdivision mechanisms, Acta Mater. 45 (1997) 3871–3886.
[20] N. Tsuji, R. Ueji, Y. Ito, Y. Saito, In-situ recrystallization of ultra-fine grains in highly strained metallic materials, Recrystallization—Fundamental aspects and relations to deformation microstructure, Proceedings of the 21st RISO International Symposium on Material Science, RISO National Laboratory, Denmark, 2000, pp. 607–616.
[21] N. Tsuji, R. Ueji, Y. Saito, Y. Minamino, Formation mechanism of ultrafine grained structure during intense straining of ferritic steels, CAMP-ISIJ 14 (2001) 494–497.
[22] N. Tsuji, T. Toyoda, Y. Minamino, Y. Koizumi, T. Yamane, M. Komatsu, M. Kiritani, Microstructural change of ultrafine-grained aluminum during high-speed plastic deformation, Mater. Sci. Eng. A 350 (2003) 108–116.