Investigation of microstructure and mechanical properties of hot worked

NiAl bronze alloy with different deformation degree

Yuting Lv, Liqiang Wang*, Yuanfei Han, Xiaoyan Xu, Weijie Lu*

State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University,
Shanghai 200240, PR China

Corresponding author. Tel: +86 21 34202641; fax: +86 21 34202749.

E-mail address: luweijie@sjtu.edu.cn, wang_liqiang@sjtu.edu.cn

Abstract:

In this study, the forged NiAl bronze (NAB) were hot rolled with the deformation degree of 40%, 60%, 80%, 90% and 95% at 850°C, respectively. Effects of rolling deformation degree on the microstructure and mechanical properties of the NAB alloy were investigated. Scanning electron microscope (SEM), electron backscatter diffraction (EBSD) and transmission electron microscope (TEM) methods were used to characterize the microstructure. The results show that α grains are refined by the dynamic recovery and recrystallization, penetration of β phase into α phase and particle-stimulated nucleation (PSN) of recrystallization during rolling. The refined grains make a main contribution to the increase of mechanical properties of rolled NAB. When the deformation degree is increased to 80%, the optimum tensile properties with ultimate strength of 861.3 ± 8.5 MPa, yield strength of 634.5 ± 7 MPa and elongation of $19.3 \pm 0.05\%$ is obtained. With further increasing the deformation degree, the strength of rolled NAB alloy increase and the elongation decrease due to the increase of work hardening effect and the formation of martensitic nano-twins.

Key words: NiAl bronze (NAB), Deformation degree, Microstructure evolution, Mechanical properties

1. Introduction

Nickel-aluminum bronze (NAB) is copper-aluminum alloy containing nickel, iron and manganese elements. The addition of alloying elements improves the mechanical properties of alloy and inhibits the formation of γ_2 phase, Al_4Cu_9 , which is deleterious to the corrosion resistance due to the high Al content [1]. NAB alloy is widely used

for marine equipment such as propeller, pump, valve etc. due to the high mechanical properties and excellent corrosion resistance [1, 2]. Typical microstructure of as-cast NAB includes α matrix phase, martensitic β phase (β' phase), iron rich κ_i , κ_{ii} , κ_{iv} and nickel rich κ_{iii} phases [3, 4]. Complex microstructure of as-cast NAB increases the possibility of galvanic corrosion which takes place in nature seawater [5, 6]. Moreover, defects such as shrinkage porosity, coarser grains are prone to appear in the cast alloys due to their poor casting properties. These defects are harmful to the mechanical and corrosion properties of components.

Hot deformation is one of the optimum approaches to refine grains and eliminate casting defects, which significantly improves the mechanical properties of casting metal material. Several plastic deformation techniques had been used to improve the mechanical properties of cast NAB alloys such as equal channel angular extrusion (ECAE) [7, 8], friction surfacing (FS) [9] and friction stir processing (FSP) [10-12] etc. It is well reported that the plastic deformation significantly improves the mechanical properties of as-cast NAB alloy [13, 14]. Ni et al. [12, 13] found that inhomogeneous microstructures were formed during FSP due to different deformation degree through stir zone. Gao et al. [7, 8] reported that the grain of the alloy was decreased with the increase of the pass number of ECAE and the mechanical properties were significantly improved after ECAE. However, it is difficult for these methods to provide direct evidence of the microstructure evolution and relation between microstructure and mechanical property with the increasing deformation degree. Forging and followed rolling is traditional hot-working technology, which is easy to control the deformation degree. Thus it can be used to investigate the variation in microstructure and mechanical properties of NAB alloy during plastic deformation.

In addition, the strengthening mechanisms of deformed NAB alloy have been investigated in many literatures [15, 16]. Ni et al. [17] found that dynamic recrystallization occurred in the stir zone during FSP. Menon et al. [16] investigated the strengthening mechanisms in NAB alloys and established the models of hot rolled NAB. Su et al. [15] found that the equiaxed α grains were produced during FSP as the local equivalent strains exceed 2.5. However, strengthening mechanisms are not

clear and need further illustrated due to the complex microstructures of NAB alloys. For example, typical microstructure of as-cast NAB alloy contains various κ phases (κ_i , κ_{ii} , κ_{iii} and κ_{iv}) with different morphologies [3, 18]. The mentioned particles can either promote or suppress recrystallization depending on their size, spacing and fraction [19]. There are few investigations to refer to the influence of the fine particles on recrystallization of NAB alloy.

In this study, the forged NAB alloy was hot rolled with deformation degree of 40%, 60%, 80%, 90%, and 95%. The effect of deformation degree on microstructure and mechanical properties of NAB alloys was investigated. The strengthening mechanisms of NAB alloy were studied and the particle-stimulated nucleation (PSN) of α grain recrystallization was also discussed.

2. Experimental Procedure

A cast ingot of Cu-9.5Al-4.2Ni-4Fe-1.2Mn NAB alloy with a dimension of $\phi 150\text{mm} \times 100\text{mm}$ was prepared by vacuum melting and then forged at 850°C . The microstructure of forged NAB alloy contains α , κ_{ii} , κ_{iv} and retained β (β') phases, as shown in Fig. 1. The rolled samples with a dimension of $75\text{mm} \times 35\text{mm} \times 30\text{mm}$ were cut from the forged ingot, heated and then equilibrated at 850°C for 20min. Hot rolling was carried out using a laboratory rolling mill and manual adjustment of the roll gap to obtain a reduction of thickness of $\sim 3\text{mm}$ on each successive pass (The reduction of thickness of the last pass is 1.5mm). The temperature of samples was maintained at the rolling temperature for 20min between each successive pass. The deformation degree was 40%, 60%, 80%, 90% and 95% respectively and the final corresponding thickness was 18, 12, 6, 3 and 1.5mm, respectively. Samples for optical microscopy were cut directly from the rolled plates and then prepared using the conventional techniques of grinding, polishing and etching. Phases structures of rolling surfaces were identified at room temperature by a D8 ADVANCE X-ray diffract meter with Cu K_α radiation. Samples for tensile test were machined from the rolled samples with the sample' axis parallel to the rolling direction, and the gauge sections of the tensile specimens were $15\text{mm} \times 4\text{mm} \times 1.5\text{mm}$. The room temperature tensile properties were tested on ZwickT1-Fr020TN. A50 with the strain rate of

10^{-3} s^{-1} . The mechanical property data for each condition were obtained by averaging four test results. The misorientation angle of the rolled sample was determined by electron backscatter diffraction (EBSD) method using a JEOL 7600-F field emission gun scanning electron microscope (SEM). A JEM-200 EX transmission electron microscope (TEM) was used to characterize the microstructures of rolled samples. For TEM sample preparation, thin plates were first cut from the rolled samples randomly and then mechanically polished to $100 \mu\text{m}$. The polished thin plates were finally twin-jet electro-polished to make electron beam transparent thin films using a solution of 33 percent nitric acid and 67 percent methanol at -25°C .

3. Experimental results

3.1 Microstructure

3.1.1 Microstructure evolution

Fig. 2 shows the rolling direction surface microstructure of the rolled NAB alloy. As can be seen, the hot deformation plays an important effect on the microstructure features. Fig. 2(a) represents the microstructure of rolled sample at deformation degree of 40%. The microstructure contains non-uniformity of coarser α phase (dark area), κ_{II} phase, κ_{IV} phase and β phase transformation product (white area), which is similar with initial microstructure (Fig. 1). At the deformation degree of 60%, elongated α grains are observed (Fig. 2(b)). With further increasing the deformation degree to above 80%, α grains show smooth boundaries (Fig. 2(c), (d) and (e)). Fig. 2(f) is an enlarged image of the β phase transformation product area inside the square frame in Fig. 2(e). As can be seen, fine α grains inside the β phase transformation product have bainite morphology. Another important feature of the rolled NAB microstructure is that volume fraction of β phase transformation product increases with the increase of deformation degree. The result is consistent with Su' study [15], which finds that plastic deformation accelerates the phase transformation $\alpha + \beta \rightarrow \beta$ during FSP and then results in the increase in volume fraction of β phase transformation product.

3.1.2 EBSD characterization

Fig. 3 shows EBSD results of rolled samples at deformation degrees of 80% and 95%, respectively. Only α grains are detected due to the difficulty of EBSD in identifying fine κ phases and martensitic β' phase. Fig. 3(a) shows the grain map of rolled sample at deformation degree of 80%. As can be seen, the α grains appear to be equiaxed and uniformly distributed. The misorientation angles distribution map of the boundary angle reveals that the fraction of low angle boundaries (below 15°) is about 60%. It is indicated that NAB experiences complete dynamic recovery and part of which is recrystallized to form new grains when the deformation degree is increased to 80%. Fig. 3(d) shows the grain map of rolled sample with deformation degree of 95%. The α grains appear to be non-uniformly distributed and some coarser grains are observed. The grain size distribution map reveals that coarser grains with grain size above $10\mu\text{m}$ forms, while grain size is uniformly distributed at deformation degree of 80% (seen from Fig. 3(c) and (f)), which indicates that some grains grows rapidly during rolling at deformation degree of 95%.

3.1.3 X-ray diffraction pattern analysis

Fig. 4 shows the X-ray diffraction patterns of rolling direction surface of NAB alloy with different deformation degrees. In comparison with smaller deformation degree (40%), the diffraction peak broadening after larger deformation, as indicated by black arrows in Fig. 4, suggests the significant reduction of grain size and occurrence of micro strain during rolling, which has been reported in former literatures [20, 21]. The main peaks of α (Cu) decreases gradually before the deformation degree of 80%. It indicates that the phase transformation of $\alpha + \beta \rightarrow \beta$ occurs during rolling, which is consistent with SEM observation results.

3.2 Tensile properties

Fig. 5 represents the uniaxial tensile stress-strain curve of rolled NAB with different deformation degrees. The tensile properties are shown in Table. 1. As can be seen from Fig. 5 and Table 1, the increase in deformation degree is very effective in improving the tensile properties of the NAB alloy. The yield strength and

the ultimate strength increase with increasing deformation degree, while the strain to failure attains a steady state before the deformation degrees of 80% and then decreases with further increasing deformation degree. It is worth mentioning that the optimum tensile property is achieved at deformation degree of 80%. The ultimate strength and the yield strength are 861.3 ± 8.5 and 634.5 ± 7 MPa, respectively, which are more than 100 and 200 MPa, respectively, higher than that of the rolled NAB with deformation degree of 60%.

4. Discussion

Based on the discussion above, rolling deformation degree has significantly influence on the microstructure and mechanical property. The optimum tensile property is achieved at the deformation degree of 80% in this study. The ultimate strength and the yield strength are more than 100 MPa and 200 MPa, respectively, higher than that of the rolled NAB with deformation degree of 60%, while similar strain to failure is obtained. Strength mechanism variation is crucial for producing better mechanical property. Therefore, it is necessary to further study the strengthening mechanism of the rolled NAB alloy.

4.1 The α grains refinement

The α grain size of rolled NAB with deformation degree of 40% and 60% are obtained based on statistical measurements of a total grain count of 200 and that of samples with deformation degrees above 80% are obtained from EBSD result, shown in Fig. 6. The grain size decreases with the increase of deformation degree. The minimum grain size is achieved at the deformation degree of 80%, which is about 3.6 μm . With further increasing deformation degree to 95%, some α grains grow and some coarser grains with grain size of above 30 μm appear, as shown in Fig. 3 (e and f). In order to find the answer to this question, detailed TEM observations of the rolled NAB at deformation degree of 95% are studied. Fig. 7 shows the TEM images of rolled NAB alloy with the deformation degree of 95%. A lot of κ_{VI} precipitations with grain size of several decade nanometers can be seen in Figs. 7 (a) and (c) and the precipitation-free area is also observed, as shown in Figs. 7(b) and (d). The

observation of large quantities of rich κ_{VI} precipitation and precipitation-free areas indicates that the fine precipitations are distributing non-uniformly in rolled NAB alloy. This fine precipitations may inhibit grain boundary mobility by Zener pinning [22], and also prevent grain growth. At precipitation-free area, the sub-grains may grow rapidly due to high stored energy obtained at the higher deformation degree. Thus some coarser α grains are obtained. Gholinia et al. [23] studied the effect of fine precipitations on the microstructure evolution of Al-Mg alloys during rolling and considered that the precipitations reduced the sub-grain size and inhibited grain boundary migration for continuous recrystallization. EBSD results show that the NAB experiences dynamic recovery, recrystallization and forms new grains at deformation degree of 80%. Therefore, the gradual increase in mechanical properties before the deformation degree of 80% is believed to be due to the grain refinement caused by dynamic recovery and recrystallization during rolling.

Fig. 8 shows the TEM images of rolled NAB alloy with deformation degree of 40%, 80% and 95%. Low dislocation density and coarser grains are observed at deformation degree of 40%, and some subgrains can also be found (Fig. 8 (a, b)). Fig. 8(c and d) shows the TEM images of the sample with deformation degree of 80%. Dislocation activity and subgrains are observed, which indicates dynamic recovery and recrystallization simultaneously occurs at deformation degree of 80%. The process of work hardening, recovery and recrystallization lead to continuous formation of low angle boundaries and dynamic recrystallization grains [24], which is consistent with the EBSD results. With further increasing deformation degree to 95%, dislocation network and high density dislocation areas are found (Fig. 8 (e and f)). The observation of large quantities of high density dislocation area indicates that work hardening make a main contribution to the strengthening of rolled NAB after deformation degree of 80%. That can explain the gradual decrease in elongation of rolled NAB alloy after the deformation degree of 80%.

In Fig. 2, block shape of β' phase is observed at deformation degree of 60%, while β' phase at deformation degree of above 80% exhibits the stream shape. It is

suggested that the β phase connects each other during rolling, which in turn splits α grains. It may be attributed to the penetration of β phase into α phase along some sub-boundaries during rolling, as indicated by dark arrows in Fig. 2(e). Su et al. [15] reported the penetration of β phase into α phase of NAB alloy during FSP and claimed that β phase penetrated into α phase along the α/α boundaries that tranversed to the major axis of the α phase. Ding et al. [25] showed that hot working led to the development of both low and high angel sub-boundary across the α grains, and then the formation of globularization α from lamellar α structure.

4.2 Particle-stimulated nucleation (PSN) of α grain recrystallization.

The microstructure of rolled NAB alloy contains relatively coarser κ_{II} particles with grain size of 1~3 μm (see Fig. 2) apart from fine κ_{VI} particles (Fig. 7(a)). The effect of large particles on promoting recrystallization via the mechanism of particle-stimulated nucleation (PSN) is well known and widely observed in other metal materials [19, 26, 27]. For lattice-diffusion control of deformation and recovery for NAB alloy, the strain rate necessary to achieve PSN of recrystallization is given by

$$\dot{\epsilon}_{PSN} \geq \frac{K_1}{r \alpha_p^2} \exp\left(-\frac{Q_D}{kT}\right), [28]$$

Where $K_1 = \frac{nGD_0\Omega}{k}$, Ω is the atom volume, G is the shear modulus, $D_{0,l}$ is the pre-exponential factor for lattice diffusion, k is the Boltzmann constant, d_p is the particle diameter, T is the temperature, and Q_D is the activation energy for lattice diffusion. For the Cu alloy used in this study, $\Omega = 1.18 \times 10^{-29} \text{m}^3$, $G = 42 \text{GPa}$, and $D_{0,l} = 0.3 \times 10^{-4} \text{m}^2 \text{s}^{-1}$, therefore, $K_1 = 0.913 \text{m}^2 \text{Ks}^{-1}$; $Q_D = 197 \text{kJmol}^{-1}$, $T = 850^\circ\text{C}$, and the average size of the second particles is $\sim 2 \mu\text{m}$. Therefore, the strain rate for PSN is $\dot{\epsilon}_{PSN} = 1.6 \times 10^{-2} \text{s}^{-1}$. In this study, average strain rates ($\bar{\epsilon}$) can be calculated by the equation

$$\bar{\epsilon} = \frac{H-h}{H} \frac{v}{\sqrt{R(H-h)}}, [29]$$

where H is initial thickness, h is final thickness of every pass, v is the roll circumferential speed (183.2mm s^{-1}) and R is the roll radius (350mm). Thus the average strain rates is 1.14, 1.6, 2.7, 4 and 5.65 s^{-1} respectively at the deformation degree of 40%, 60%, 80%, 90% and 95%, which is at least one order of magnitude higher than that of PSN strain rate ($\dot{\epsilon}_{PSN}$). Therefore, PSN is a plausible mechanism for recrystallization in the primary α grains of NAB alloy around second particles. The current result is consistent with Ohishi' study [28], which showed that PSN of NAB alloy occurred at sire zone/thermo-mechanically affected zone interface around κ_{II} particles during friction stir processing.

4.3 Martensite twins

Fig. 9 shows TEM images and electron diffraction pattern (inset in Fig. 9 (a and c)) obtained at deformation degree of 80% and 90%. Martensite phase can be indentified from selected diffraction pattern, which has a twin substructure with average thickness of $\sim 15\text{nm}$. The martensite locates at grains boundaries around large quantities of high density dislocation, which is significant different from martensite phase of casting NAB alloy [3]. The twins may contribute to strenthening of the rolled NAB alloy by acting as a grain boundary and blocking the lattice dislocation motion [30]. It is worth mentioning that less martensite twins are found at the deformation degree of 40%. Therefore, that may be another reason for the significant improvement of mechanical properties of rolled NAB alloy as increasing deformation degree from 60% to 80%.

As is well known, twinning and dislcotion slip are two major and competing modes of plastic deformation in metals and alloys [20]. During plastic deformation the operative mode is determined by the conditions of deformation and the intrinsic properties of the deformed alloy [20]. Stacking fault energy (SFE) is one of the most important parameters. The alloys with low SFE have a higher twinning tendency than these with high SFE [33]. The addition of Al in Cu alloy reduces the SFE and changes the energy path for twinning, which has been reported by many investigation [20, 31, 32]. Zhang et al. [33, 34] studied the effect of stacking faults on the strength of ultra-fine grained Cu-Al alloy and pointed out that lower SFE made a main

contribution to the higher strength of the Cu-Al alloy. Moreover, high density dislocations are found at the twin boundaries (see Fig. 9 (b and d)), which is typical deformation twins [33]. Therefore, we believe that dislocations accumulate gradually at grain boundaries with the increase of deformation degree, which promote the formation of twins after deformation degree of 80% due to lower SFE. That is the reason why more martensitic twins are found after deformation degree of 80%.

Based on the discussed above, we believe that the dynamic recovery and recrystallization, penetration of β phase into α phase and particle-stimulated nucleation (PSN) of recrystallization during rolling lead to the refinement of α grains, which make a main contribution to the increase of mechanical properties before deformation degree of 80%. It is well known that the yield strength of polycrystalline materials increase with decreasing grain size, as described by Hall-Petch relation. Therefore, we believe that yield strength is a better indicator of the deformation accumulation effect before deformation degree of 80%. With further increasing the deformation degree, the α grain size is hardly influenced by deformation degree. The work hardening effect and the increase of martensitic nano-twins are proven to be the main reason responsible for the strength of NAB alloy, which also lead to the significantly decrease in elongation of rolled NAB alloy. In this situation, the ultimate strength may be a better indicator of the deformation accumulation effect.

5. Conclusions

In this study, the microstructure and mechanical properties of hot rolled NiAl bronze alloy with different deformation degree were investigated. According to the experimental findings, the following conclusions can be drawn from this work.

(1) Deformation degree plays an important effect on the microstructure and mechanical properties of NAB alloy. The tensile properties of rolled NAB alloy increase with the increase of deformation degree before 80% due to the refinement of grains. With further increasing deformation degree, work hardening and formation of martensitic nano-twins make a main contribution to the strength of NAB alloy, which also lead to the significantly decrease in elongation of rolled NAB alloy.

(2) The optimum mechanical properties with ultimate strength of 861.3 ± 8.5 MPa,

yield strength of 634.5 ± 7 MPa and total elongation of $19.3 \pm 0.05\%$ are achieved at deformation degree of 80%.

(3) Particle-stimulated nucleation (PSN) of recrystallization of NAB alloy occurs during rolling due to higher strain rate and presence of coarser second particles.

(4) The mechanisms of refinement of α grains mainly contain dynamic recovery and recrystallization, penetration of β phase into α phase and particle-stimulated nucleation (PSN) of recrystallization.

Acknowledgements

Financial support of this research provided by 973 Program under Grant No: 2014CB046701, China Postdoctoral Science Foundation Grant No: 2014M561471. National Science Foundation under Grant No: 51302168, Shanghai Natural Science Foundation under Grant No: 12ZR1445500, New Teachers' Fund for Doctor Stations, Ministry of Education under Grant No: 20120073120007.

Reference

- [1] E.A. Culpan, G. Rose. J. Mater. Sci. 13 (1978) 1647-1657.
- [2] A. Jahanafrooz, F. Hasan, G.W. Lorrmer, and N. Ridley. Metall. Mater. Trans. A. 14A (1982) 1951-1956.
- [3] F. Hasan A. Jahanafrooz, G.W. Lorimer, and N. Ridley. Metall. Mater. Trans. A. 13 (1982) 1337-1345.
- [4] Y. Liu, J. Mazumder and K. Shibata. Acta metall. mater. 42 (1994) 1755-1762.
- [5] J.A. Wharton, R.C. Barik, G. Kear, R.J.K. Wood, K.R. Stokes, F.C. Walsh. Corros. Sci. 47 (2005) 3336-3367.
- [6] S. Fonlupt, B. Bayle, D. Delafosse, J.L. Heuze. Corros.Sci. 47 (2005) 2792-2806.
- [7] L.L. Gao, X.H. Cheng. Wear. 265 (2008) 986-991.
- [8] L.L. Gao, X. Cheng. Mater. Des. (29) 2008 904-908.
- [9] S. Hanke, A. Fischer, M. Beyer, J. Santos. Wear. 273 (2011) 32-37.
- [10] O. Kellchiroand and R.M. Terry. Metall. mater. Trans A. 35A (2004) 2951-2960.
- [11] S. Swaminathan, O. Keiichiro, Z. Alexander P, F. Christian B, L. Blair, M. Murray W and R.M. Terry. Metall. Mater. Trans. A. 41 (2009) 631-640.
- [12] D.R. Ni, B.L. Xiao, Z.Y. Ma, Y.X. Qiao, Y.G. Zheng. Corros. Sci. 52 (2010)

1610-1617.

- [13] D.R. Ni, P. Xue, Z.Y. Ma. Metall. Mater. Trans.A. 42 (2011) 2125-2135.
- [14] O. Kellchiro and M. Terry R. 36A (2004) 1575-1585.
- [15] J.Q. Su, S. Swaminathan, S.K. Menon, T.R. Mcnelley. Metall. Mater. Trans. A. 42 (2011) 2420-2430.
- [16] K.M. Sarath, A.P. Frank, P. R. Brian, O. Kellchiro, S. Srinivasan and R.M. Terry. Metall. Mater. Trans. A. 43 (2012) 3687-3702.
- [17] D.R. Ni, P. Xue, D. Wang, B.L. Xiao, Z.Y. Ma. Mater. Sci. Eng. A. 524 (2009) 119-128.
- [18] Y. Liu, M.E. Mochel, J. Mazumer and K. Shibata. Acta metll.mater. 42 (1994) 1763-1768.
- [19] J.D. Robson, D.T. Henry, B. Davis. Acta. Mater. 57 (2009) 2739-2747.
- [20] K. Youssef, M. Sakaliyska, H. Bahmanpour, R. Scattergood, C. Koch. Acta. Mater. 59 (2011) 5758-5764.
- [21] X.X. Wu, X.Y. San, X.G. Liang, Y.L. Gong, X.K. Zhu. Mater. Des. 47 (2013) 372-376
- [22] F.J. Humphreys and M.G. Ardakani. Acta. Mater. 44 (1996) 2717-2727.
- [23] A. Gholinia, F.J. Humphreys, P.B. Prangnell. Acta. Mater. 50 (2002) 4461-4476.
- [24] Y.F. Han, W.D. Zeng, Y.L. Qi, Y.Q. Zhao. Mater. Sci. Eng. A. 528 (2011) 8410-8416.
- [25] R. Ding, Z.X. Guo, A. Wilson. Mater. Sci. Eng. A. 365 (2002) 172-179.
- [26] J.D. Robson, D.T. Henry, B. Davis. Mater. Sci. Eng. A. 528 (2011) 4239-4247.
- [27] W. Xu, M. Ferry, J. Cairney, F. Humphreys. Acta. Mater. 55 (2007) 5157-5167.
- [28] O. Kellhiro, Z.P. Alexander, and R.M. Terry Metall. Mater. Trans A. 37A (2006) 2239-2251.
- [29] S.Q. Zhu, H.G. Yan, J.H. Chen, Y.Z. Wu, J.Z. Liu, Tian J. Scripta. Mater. 63 (2010) 985-988.
- [30] L. Lu, X. Chen, X. Huang, K. Lu. Science. 323 (2009). 607-610.
- [31] X.H. An, Q.Y. Lin, S.D. Wu, Z. F. Zhang, R. B. Figueiredo. Scripta. Mater. 64 (2011) 954-957.

- [32] X.Y. San, X.G. Liang, L.P. Cheng, L. Shen, X.K. Zhu. Trans. Nonferous Met. Soc. china. 22 (2012) 819-824.
- [33] Y. Zhang, N.R. Tao, K. Lu. Acta. Mater. 59 (2011) 6048-6058.
- [34] Y. Zhang, N.R. Tao, K. Lu. Scripta. Mater. 60 (2009) 211-213.

Fig. 1. Initial microstructures of forged NAB alloy.

Fig. 2. SEM images of rolled samples with different deformation degrees: (a) 40%, (b) 60 %, (c) 80%, (d) 90%, (e) 95% and (f) Magnified image of area inside square frame shown in Fig. 2(e).

Fig. 3. EBSD results of rolled NAB: (a) EBSD map at the deformation degree of 80%, (b) Misorientation distribution of the boundary angle of Fig. 3 (a), (c) Grain size distribution of Fig. 3 (a), (d) EBSD map at deformation degree of 95%, (e) Misorientation distribution of the boundary angle of Fig. 3 (d), (f) Grain size distribution of Fig. 3 (d).

Fig. 4. X-ray diffraction patterns of rolled NAB with different deformation degrees.

Fig. 5. Engineering stress-strain curves of the rolled NAB alloy with different deformation degrees.

Fig. 6. The α grain size variations as a function of deformation degrees.

Fig. 7. TEM images of the rolled NAB at the deformation degree of 95%. (a) and (c) Rich precipitation areas, (b) and (d) Precipitation-free areas.

Fig. 8. TEM images of the rolled NAB with different deformation degree. (a) and (b) 40%, (c) and (d) 80%, (e) and (f) 95%.

Fig. 9. TEM images of rolled NAB martensite twins. (a) Deformation degree of 80% and selected area diffraction pattern (upper right inset), (b) Magnified image of area inside square frame shown in Fig. 9(a), (c) Deformation degree of 90% and and selected area diffraction pattern (upper right inset), (d) Magnified image of the area inside square frame shown in Fig. 9(c).

Table 1. Tensile properties of rolled NAB alloy.

	40%	60%	80%	90%	95%
YS (MPa)	407±3.4	426±13	634.5±7	716±24	842±21
TS (MPa)	728.1±4.6	761±14	861.3±8.5	957.3±28	1134.7±23.5
EL (%)	18.4±1.3	19.8±2	19.3±0.05	8.7±0.4	5.13±0.2