

Effects of Strain Increment on Grain Refining in Pure Aluminum during Room Temperature Multiple Directional Forging

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Microstructure evolution of pure aluminums with two different purities was investigated during multi-directional forging (MDF) process at room temperature. Both of purity and strain increment ($\Delta\epsilon$) affect the grain refining rate in the aluminum during MDF. The flow stress during MDF increases with increasing of $\Delta\epsilon$. In low $\Delta\epsilon$ region ($\Delta\epsilon < 0.5$), the value of maximum flow stress for every pass is saturated with increasing cumulative strain. Electron backscatter diffraction measurement of MDFed 4N Al with $\Delta\epsilon = 0.5$ points out that the development of micro-shear bands in various directions and their intersections are important to develop the high angle grain boundaries (HAGBs). On the other hand, the saturation behavior of maximum flow stress is hardly observed in MDF with $\Delta\epsilon = 0.8$ in 2N Al. The grain refining is accelerated by increasing of $\Delta\epsilon$. Many HAGBs are formed heterogeneously in the early stage of MDF with $\Delta\epsilon = 0.8$. The $\Delta\epsilon$ value is significantly important to the formation of HAGBs during MDF process in pure aluminum. Additionally, it is suggested that the nucleation sites of HAGBs in MDF of the aluminum change from “cross section points of dense dislocation walls and micro-share bands” to “boundaries of dense dislocation walls and micro-share bands” with increasing $\Delta\epsilon$.

Keywords: severe plastic deformation, multi-directional forging, grain refining, pure aluminum, strain increment.

1. Introduction

Ultrafine grained materials have been produced by various severe plastic deformation (SPD) methods at low temperature below the half of melting point, $0.5T_m$, and these ultrafine grained materials are achieved excellent strength (hardness, yield stress and so on) [1, 2]. Multiple directional forging (MDF) method is one of the SPD processes. This method is basically simple cycle compression and the compression axis is rotated by 90 degree in each pass. This method does not require any special equipment. Additionally, in MDF method, the stress-strain information can be investigated during manufacturing. MDF process has been applied to copper, α -iron and 1100 aluminum since 1950s. Recently, MDF process was used to 304 steel, aluminum alloys and magnesium alloys in combination with the decreasing temperature conditions [3-5] and these MDFed alloys exhibit high strength and high ductility. However, from the viewpoints of microstructure evolution during severe plastic deformation, the understanding of grain refining processes has not been enough yet. The aims of this study is to investigate the effects of purity and strain increment ($\Delta\epsilon$) on microstructural development during MDF at room temperature, characterize deformation microstructure during MDF, and discuss the grain refining mechanism of pure aluminum during this process.

2. Experimental Procedures

The materials used in this investigation were two kinds of pure aluminum with different purity, 99+% (2N) and 99.99% (4N). They were solution-treated in air at 800 K for 2 h and then water quenched.

Average grain diameters of these specimens after this solution treatment are about 300 μm for 2N aluminum and over 500 μm for 4N aluminum. Some coarse precipitates with diameter of 1 ~ 2 μm are observed on grain boundaries in 2N aluminum after the solution treatment, but such precipitates are hardly observed in 4N aluminum before and after the solution treatment. MDF processes were performed at room temperature in air atmosphere at an initial strain-rate of $3 \times 10^{-3} \text{ s}^{-1}$. Strain increments $\Delta\epsilon$ were ranging from 0.2 to 0.8. The forging axis was changed by 90 degrees for every pass. Specimens were machined in rectangular shapes and their height-length-width ratios were 1.22 : 1.10 : 1 for $\Delta\epsilon = 0.2$, 1.65 : 1.28 : 1 for $\Delta\epsilon = 0.5$, and 2.22 : 1.49 : 1 for $\Delta\epsilon = 0.8$, respectively. A height-length-width ratio can be calculated and was kept constant during MDF processes with each strain increment. Microstructures of MDFed specimens were observed and analyzed by electron backscatter diffraction pattern (EBSD) measurements in a field-emission type scanning electron microscope (FE-SEM) and transmission electron microscope (TEM) at an acceleration voltage of 200 kV.

3. Results and Discussion

3.1 Stress-Cumulative strain Curves

Typical true stress-cumulative strain ($\sigma - \Sigma\Delta\epsilon$) curves of 2N aluminum with $\Delta\epsilon = 0.5$ during room temperature MDF are shown in Fig.1. The clear normal work hardening behavior is observed in the first pass of MDF. On the other hand, the temporary decrease of the flow stress (stress drop) is observed in parts of MDF passes. This stress drop behavior is already observed in the second pass. The amount of the stress drop is ranging from several to ten MPa. Such clear stress drop behavior is not observed during MDF working to pure copper at low and room temperatures [6]. Softening by the dynamic and/or the static recrystallization cannot be considered to explain this behavior due to the following two reasons; (1) No clear evidence of the recrystallization is observed in the microstructures of interrupted MDFed samples at the stress drop in 2N aluminum, and (2) the stress drop behavior is not observed during uniaxial intermittent compression passes with $\Delta\epsilon = 0.5$ in 4N aluminum.

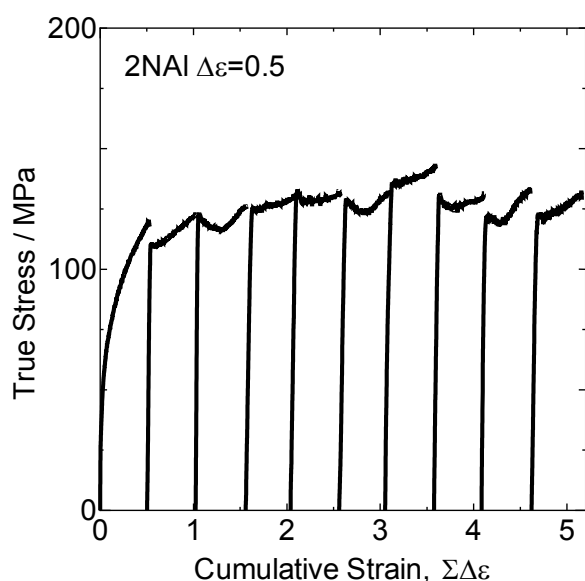


Fig.1 Cumulative Strain ($\Sigma\Delta\epsilon$)-Stress curves of 2N aluminum during MDF at room temperature with the strain increment ($\Delta\epsilon$) of 0.5.

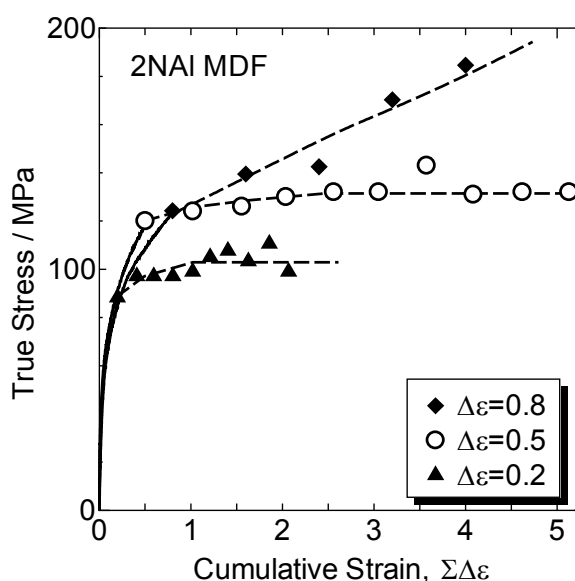


Fig.2 Maximum flow stresses plotted as a function of cumulative strain during room temperature MDF in 2N aluminum with $\Delta\epsilon = 0.2, 0.5$ and 0.8 .

3.2 Effects of strain increment and purity on the flow stress

The maximum flow stresses of each pass in 2N aluminums with three kinds of strain increments are plotted as the function of cumulative strain in Fig. 2. The stress-strain curves of the first pass in each sample are shown in this figure. The strength increases in the early stage of the MDF and reaches constant up to the cumulative strain $\Sigma\Delta\epsilon$ of 2. Additionally, the saturated flow stress increases with increasing the stress increment, $\Delta\epsilon$. This saturation behavior is observed in the low $\Delta\epsilon$ values ($\Delta\epsilon < 0.5$). On the other hand, clear saturation behavior was not observed when the value of strain increment increases to $\Delta\epsilon = 0.8$, at least less than $\Sigma\Delta\epsilon$ of 4.8 (after 6 Passes). Similar trend is observed in 4N aluminum, however, the saturation behavior of flow stress is observed with $\Delta\epsilon = 0.8$.

According to P.E. Armstrong et al, the saturated flow stress σ_{sfs} depends on $\Delta\epsilon$ and purity and can be plotted as a function of $\Delta\epsilon$ according to the following equation [7]

$$\frac{\sigma_{sfs}}{\sigma_s} = A|\Delta\epsilon|^n \quad (1)$$

where σ_s is steady-state flow stress under unidirectional compression, A and n are material constants.

They have reported that the same relationship as Eq. (1) is observed during cyclic testing in copper, α -Fe and 1100 commercial aluminum [8-10] and the n value of 0.19 is calculated in MDF of 1100 aluminum with $\Delta\epsilon$ up to 0.33 [7]. The relationships between strain increment $\Delta\epsilon$ and saturated flow stress σ_{sfs} in 2N and 4N aluminums are shown in Fig. 3. The saturated flow stress decreases with increasing purity and the similar n value to 1100 aluminum is observed in low $\Delta\epsilon$ region ($\Delta\epsilon < 0.5$). However, the data point of $\Delta\epsilon = 0.8$ of 4N Al deviates from the extrapolation of the relationship described by Eq. (1). Additionally, the clear saturation behavior itself is not observed in 2N aluminum with $\Delta\epsilon = 0.8$. This result indicates that the power law relationship between $\Delta\epsilon$ and σ_{sfs} can be described during MDF within limited $\Delta\epsilon$ region only, lower than 0.5. The flow stress and σ_{sfs} are also affected by purity. The difference of the flow stresses between 2N and 4N aluminum with $\Delta\epsilon < 0.5$ is about 20 MPa. This value is similar to the difference of ultimate tensile strength of cold worked pure aluminums, between 99.60~99.45% aluminum and 99.99% aluminum [11]. Some coarse precipitates are formed in 2N aluminum after solution treatment at 800 K for 2h. However, the number density of these precipitates is quite low thereby the precipitation hardening is hardly considered in 2N aluminum during MDF. On the other hand, this heat-treatment temperature is high enough to consider the rest of solute atoms to be in solid solutions.

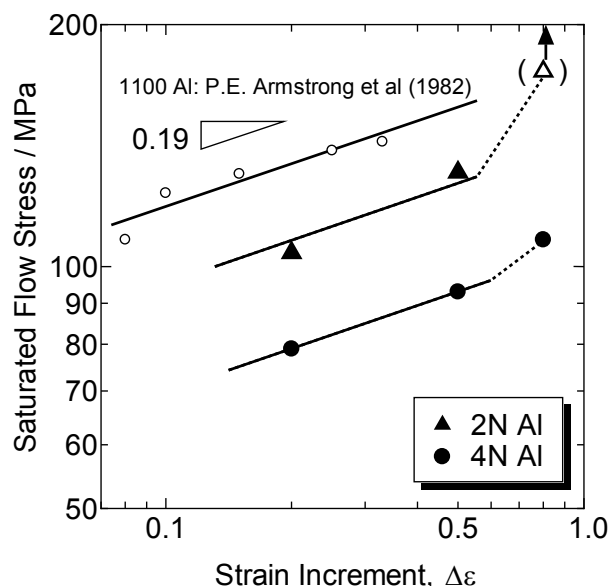


Fig.3 Strain increment ($\Delta\epsilon$) dependence of saturated flow stress (σ_{sfs}) of 2N and 4N aluminum. Data points of 1100 aluminum have been reported in [7].

3.3 Microstructure Evolution during MDF

Figure 4 shows a series of grain boundary mappings in 2N aluminum after various MDF passes with $\Delta\epsilon = 0.8$. It can be recognized that the high angle grain boundaries (HAGBs) are already formed after 1 pass of MDF (same as the simple single compression test) within grains (Fig 4(a)). The

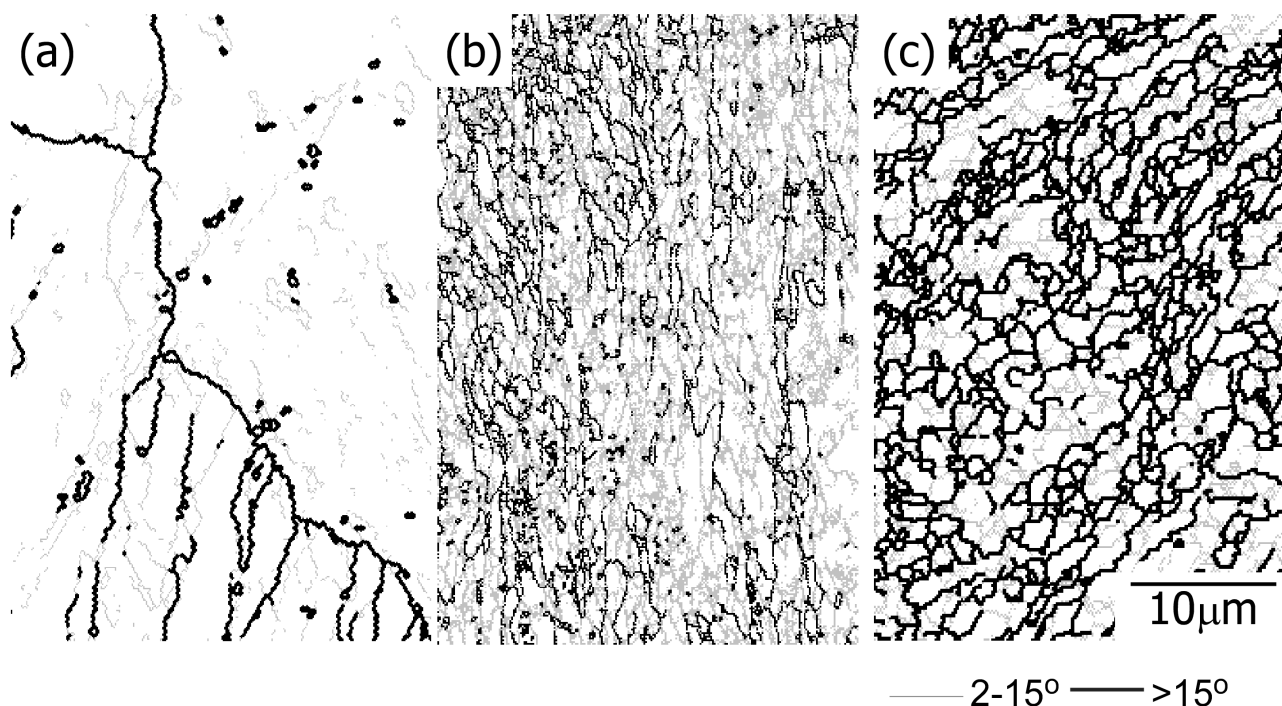


Fig.4 Grain boundary mappings of MDFed 2N aluminum at various cumulative strains.

The strain increment $\Delta\epsilon$ is 0.8 and last compression axis is vertical direction.

(a) $\Sigma\Delta\epsilon = 0.8$ (1 Pass), (b) $\Sigma\Delta\epsilon = 2.4$ (3 Pass), (c) $\Sigma\Delta\epsilon \sim 4.2$ (interrupt during 6 pass)

distribution of HAGBs is quite heterogeneous in Fig. 4(a). Some grains have many HAGBs, but HAGBs are hardly observed in the other grains. These HAGBs are elongated to one direction in each grain and roughly estimated interspacing between these HAGBs is several micrometers. Large and gradual crystalline rotation is observed in whole of initial grains without HAGBs. Although photos are not shown in this paper, transmission electron microscopic observations revealed that the elongated low grain boundaries (LAGBs) are observed within these grains instead of elongated HAGBs, having similar interspacing between boundaries. These boundaries are considered as the dense dislocation walls (DDWs) [12] and/or the boundaries of microshare bands (called as S-bands, too) [6, 13]. From the EBSD results of Figs. 4 and 5, it is considered that the DDWs and/or microshare bands can develop from LAGBs to HAGBs after 1 pass of MDF when the strain increment $\Delta\epsilon$ is large enough. At the high cumulative strain of $\Sigma\Delta\epsilon$, the fine equiaxed grains which are surrounded with HAGBs are observed. However, this grain refining occurs heterogeneously during MDF with $\Delta\epsilon = 0.8$ as well as the formation of HAGBs within initial grains. This trend is still observed after 7 pass of MDF, $\Sigma\Delta\epsilon = 5.6$. Most of the new grains have an elliptic shape, but their major direction takes random orientations because of the 90 degree rotating of work directions in every MDF passes.

The formation of HAGBs was significantly suppressed by the decreasing of $\Delta\epsilon$. Figure 5 shows the grain boundary mapping of 2N aluminum after 2 pass of MDF with $\Delta\epsilon = 0.5$ ($\Sigma\Delta\epsilon = 1.0$). Although higher cumulative strain $\Sigma\Delta\epsilon$ is given to this sample than that of 1 pass MDF condition with $\Delta\epsilon = 0.8$ (Fig. 4(a)), no new HAGBs are formed within grains. This result indicates that the strain increment is significantly important for the formation of HAGBs. In other words, a certain degree of strain increment is required to form HAGBs efficiently.

The purity also affects the formation of HAGBs. The grain boundary mapping of 4N aluminum after 6 passed of MDF with $\Delta\epsilon = 0.5$ ($\Sigma\Delta\epsilon = 3.0$) is shown in Fig. 6. DDWs and/or microshare bands

elongated to different directions are observed in it as well as Fig. 5. The most of boundaries are still LAGBs and the interspacing between them are larger than that of 2N aluminum at $\Sigma\Delta\epsilon = 0.8$ with $\Delta\epsilon = 0.8$ (1 passed MDF, Fig. 4(a)). The HAGBs are partly formed at the intersections of these microshare bands and the number of HAGBs increases with increasing MDF passes.

It is considered that the dislocation annihilation in 4N aluminum is easier than that in 2N aluminum because no HAGBs are observed within grains in 4N aluminum after 1 pass MDFed sample with $\Delta\epsilon = 0.8$. Under the conditions of low purity and high strain increment of MDF, the accumulation of dislocations on DDWs and microshare bands occurs effectively, thereby the DDWs and the boundaries of the microshare bands can increase their misorientation angle, in other words, they can develop from LAGBs to HAGBs in early MDF passes. On the other hand, when high purity and/or low strain increment are selected in MDF process, the intersection of DDWs or microshare bands is important for the nucleation of HAGBs.

In MDF process, fine equiaxial grains are formed at high cumulative strain. The grain sizes including subgrain boundaries in 4N aluminum around $\Sigma\Delta\epsilon = 5.0$ are about 3~4 μm for $\Delta\epsilon = 0.5$ and 2~3 μm for $\Delta\epsilon = 0.8$, respectively. The grain size of MDFed 2N aluminum in grain refining area is about 1~2 μm after 7 passes of MDF ($\Sigma\Delta\epsilon = 5.6$) with $\Delta\epsilon = 0.8$. Both of the purity and the strain increment affect to determine the final refined grain size formed at large cumulative strain, but the rate of grain refinement is significantly affected by the value of the strain increment.

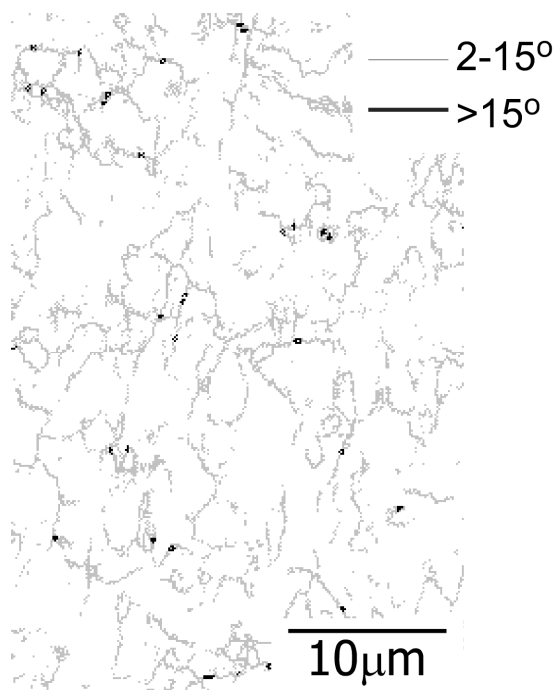


Fig.5 Grain boundary mappings of 2N aluminum after 2 passes of MDF with $\Delta\epsilon = 0.5$ ($\Sigma\Delta\epsilon = 1.0$). Last compression axis is vertical direction.

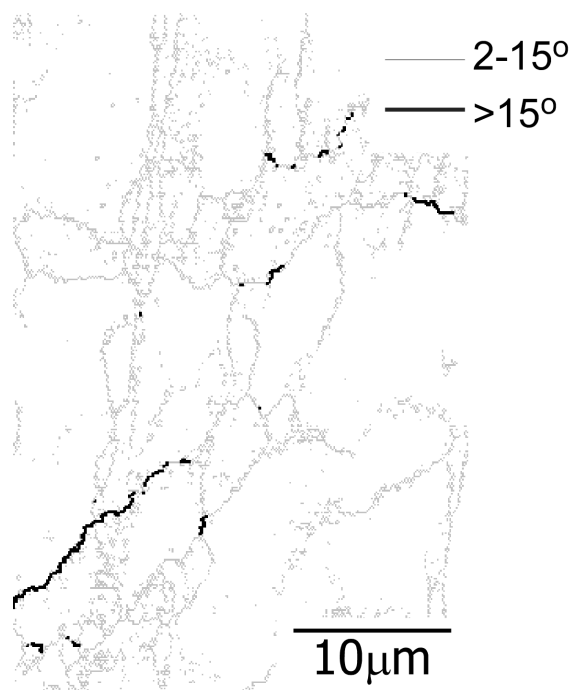


Fig.6 Grain boundary mappings of 4N aluminum after 6 passes of MDF with $\Delta\epsilon = 0.5$ ($\Sigma\Delta\epsilon = 3.0$). Last compression axis is vertical direction.

4. Summary

The effects of purity and microstructural evolution during MDF in aluminum were investigated and summarized as follows;

1. The saturated flow stress σ_{sfs} during MDF decreases with increasing purity. The value of σ_{sfs} depends on the stress increment of $\Delta\epsilon$ and the power-law relationship is observed in the low $\Delta\epsilon$ region. However, the data point of σ_{sfs} with $\Delta\epsilon = 0.8$ deviate from this relationship. Especially, in 2N aluminum, the saturation behavior of the flow stress is not observed during MDF with $\Delta\epsilon = 0.8$.
2. The formation of HAGBs during MDF process depends on the strain increment, $\Delta\epsilon$. Many HAGBs are formed heterogeneously within grains in 2N aluminum after 1 pass of MDF with $\Delta\epsilon = 0.8$. On the contrary, the formation of HAGBs is hardly observed after 2 passes of MDFed samples with $\Delta\epsilon = 0.5$.
3. The nucleation sites of HAGBs during MDF with low $\Delta\epsilon$ in 4N aluminum are the intersections of microshare bands, which are developed with various directions within grains. On the other hand, DDWs and boundaries of microshare bands become HAGBs heterogeneously with high $\Delta\epsilon$ of 0.8 in 2N aluminum. The strain increment significantly affects the grain refining process during MDF in pure aluminum.

Acknowledgments

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References

- [1] *Nanomaterials by severe plastic deformation*, ed. by Z. Horita, Trans Tech Publications, (2005)
- [2] R. Z. Valiev, R. K. Islamgaliev and I. V. Alexandrov: Prog. Mater. Sci., 45(2000), 130-189
- [3] A. Belyakov, T. Sakai, H. Miura and R. Kabyshev, T. Tsuzaki: Acta Mater., 50(2002), 1547-1557
- [4] O. Sitdikov, T. Sakai and A. Goloborodko: H. Miura, Scr. Mater., 51(2001), 175-179
- [5] J. Xing, H. Soda, X. Yang, H. Miura and T. Sakai: Mater. Trans., 46(2005), 1646-1650
- [6] T. Sakai, H. Miura and X. Yang: Mater. Sci. Eng., A499(2009) 2-6
- [7] P. E. Armstrong, J. E. Hockett and O. D. Sherby: J. Mech. Phys. Solid, 30(1982), 37-58
- [8] L. F. Coffin and J. F. Tavernelli: Trans. AIME, 215(1959), 794-807
- [9] C. E. Feltner and C. Laird: Acta Metall., 15(1967), 1621-1632
- [10] H. Abdel-Raouf, P. P. Benham and A. Plumtree: Can. Metall. Quart., 10(1971), 87-95
- [11] N. A. Belov, A. A. Aksenov and D. G. Eskin: *Iron in Aluminum Alloys: Impurity and Alloying Element*, (Taylor & Francis, 2002), pp.161
- [12] B. Bay, N. Hansen and D. Kuhlmann-Wilsdorf: Mater. Sci. Eng., A113(1989), 385-397
- [13] G. I. Rosen, D. J. Jensen, D. A. Hughes and N. Hansen: Acta Metall. Mater., 45(1995), 2563-2579