

Mechanical properties of an Al-Mg-Sc alloy subjected to intense plastic straining

R. Kaibyshev^{1a}, E. Avtokratova^{2b}, O. Sitdikov^{2c}

¹ Belgorod State University, Belgorod 308034, Russia

² Institute for Metals Superplasticity Problems, Khalturina 39, Ufa 450001, Russia

^a rustam_kaibyshev@bsu.edu.ru; ^b lena@imsp.da.ru, ^c sitdikov@nm.ru

Keywords: mechanical properties, aluminum alloy, ECAP, rolling, UFG structure.

Abstract. Effect of intense plastic straining on rollability and service properties of an Al-6%Mg-0.3%Sc alloy was examined. Ultrafine-grained structure (UFG) was produced by equal-channel angular pressing (ECAP) to a strain of ~ 8 at a temperature of 325°C. The formation of UFG structure resulted in increase in the yield stress from 223 MPa to 285 MPa and ultimate stress from 350 MPa to 389 MPa in comparison with initial hot extruded condition. Total elongation slightly decreased from 33% to 29%. After ECAP, the material was subjected to cold and isothermal warm rolling. The formation of UFG structure resulted in enhanced rollability of the present alloy at room temperature. Cold rolling with high reduction provides the development of heavily deformed microstructure with high dislocation density, while the isothermal warm rolling does not remarkably affect the microstructure produced by ECAP. The mechanical properties after ECAP and ECAP with subsequent isothermal rolling were roughly similar. In contrast, cold rolling to the same strain resulted in significant increase of yield stress (495 MPa) and ultimate stress (536 MPa). Total elongation attained was 13%.

Introduction

Recent work [1] showed that there is a great potential for extraordinary improvements in the chemical, physical and mechanical properties of aluminium-based alloys by refining their grain structure through severe deformation to the submicron or nanocrystalline level (grain size less than 1 and 0.1 μm , comparatively). Methods based on intense plastic straining (IPS) are most attractive for this goal [1, 2]. Most popular for commercial application is ECAP [e.g. 1-3], which can be used for intense deformation at ambient and elevated temperatures to produce bulk ultrafine-grained materials. However, regarding wrought Al alloys, which are much in demand for cold- or warm-rolled products [3], manufacturing of thin sheets with UFG structure may be also very important for commercial use. In this way, a technique that includes combination of ECAP and subsequent cold or warm rolling would be a very promising technical procedure with great commercial potential. Nowadays, the role of such deformation scheme in producing sheet materials with UFG structure and enhanced mechanical properties is, however, quite poor specified in literature.

Complex studies of the Al-Mg-Sc based alloys showed that these alloys are advanced structural materials with enhanced service properties [4]. In particular, these alloys have a high strength and also an excellent corrosion resistance and good weldability that make them very promising candidates for use in automotive, aviation and space industries. The most serious deficiency of the Al-Mg-Sc alloys with the Mg content $\geq 3\text{wt}\%$ is however a poor technological plasticity and, especially, a limited rollability at ambient and intermediate temperatures [4, 5]. Actually, it is possible to produce thin Al-Mg-Sc sheets by cold rolling of original coarse-grained alloy, but this procedure requests a lot of cutting operations in order to eliminate many cracks at the edge of the rolled samples [5]. Owing to a strong localization of plastic flow upon cold working, the first surface cracks start to appear in these materials already after the engineering strains of about 15%. In contrast, it can be expected that the strain localization will be minimized, when the original fine-grained state will be achieved before rolling.

The aim of the present work is to evaluate the potential for improvement of rollability of the Al-6%Mg-0.3%Sc alloy by refining their grain structure to the submicron level through ECAP at $T \sim 0.6 T_m$. Upon this consideration, the behavior of the alloy with initial submicrometer grain structure is investigated during cold rolling and compared with that during rolling at preliminary ECAP temperature. The influence of cold and warm deformation on structure and mechanical properties of material with UFG structure is analyzed and discussed in detail.

Experimental

The alloy used had the following chemical composition (in mass %): 6Mg, 0.4Mn, 0.3Sc, 0.2Si, 0.1Fe and the balance Al. The as-extruded rod was fabricated at the Kamensk-Uralsk Metallurgical Works (Russia) by casting and hot extrusion that was performed at 390°C to a strain of about 0.7, followed by annealing at 400°C for 1h. After extrusion and annealing, the alloy was composed of a non-uniform partially recrystallized microstructure with a bimodal distribution of the grain size, namely, coarse elongated grains lying parallel to the extrusion axis, and fine equiaxed grains in their mantle regions [6-9]. The size of coarse grains was ~ 170 and ~ 70 μm in longitudinal and transverse directions, respectively. The fine grains had the average size of 4.4 μm and the volume fraction of 35%¹. Samples for ECAP were machined parallel to the extrusion axis into square plates having dimensions of 125×125×25 mm³. ECAP was carried out at 325°C using a die with a rectangular cross-section of 125×25 mm² and channel inner and outer angles of $\Phi=90^\circ$ and $\psi=0^\circ$. These angles led to a strain of ~ 1 in each ECAP passage. The samples were pressed to 8 passes through route D (Bcz), i.e. the plate was rotated by 90° in the same sense around the normal axis to the plate between each pass [6]. The ECAP temperature of 325°C was chosen for the current material as the optimum one at that the microstructure with the largest fraction of fine grains and the smallest grain size is acquired [7, 8]. Thus, after ECAP, the alloy had an almost uniform UFG structure with the grain size of about 1 μm and the volume fraction of about 86%. (Fig. 1). Besides, some remnant parts of original grains were present in this structure.

Plates with dimensions of 60×25×12 and 60×12×10 mm³ were machined from the ECAE-processed billets for subsequent warm and cold rolling, respectively. The warm rolling was carried out under isothermal conditions at 325°C with the engineering strain of $\sim 10\%$ per a pass, while the cold rolling was performed at ambient temperature with the strain per a pass of $\sim 5\%$. In both cases, the rolling direction (RD) coincided with the last ECAP direction (PD). Deformed microstructures were examined in the sections parallel to the normal and longitudinal planes of the rolled samples using optical microscopy and TEM. Tensile specimens with a 6 mm gauge length and 3 mm gauge width were machined from the sheets with a gauge length parallel to RD. Details of microstructural characterization and mechanical testing techniques of the thin sheets are reported elsewhere [5-9].

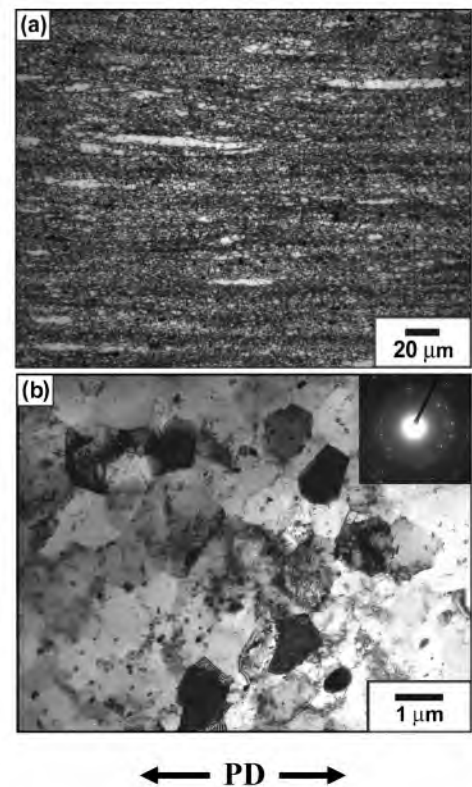


Fig. 1. Typical microstructure of Al-6%Mg-0.3%Sc alloy after ECAP to $\epsilon=8$; $T=325^\circ\text{C}$. PD is the last pressing direction.

Results and Discussion

Rollability of the UFG Al-6%Mg-0.3%Sc alloy. There were not any problems to roll the samples with the UFG structure introduced by ECAP. Both at room temperature and at $T=325^{\circ}\text{C}$ the samples

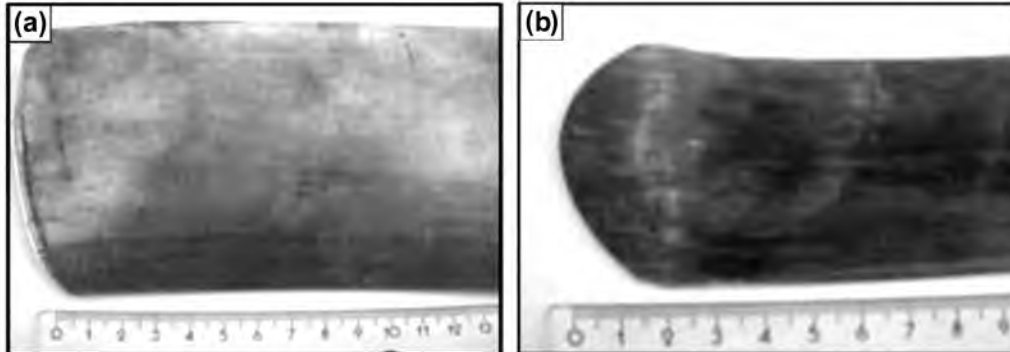


Fig. 2. Typical photographs of thin sheets produced by (a) cold and (b) isothermal warm rolling of Al-6%Mg-0.3%Sc alloy with UFG structure obtained by ECAP.

were successfully rolled to a total reduction of about 80-85% without cracking. The typical thin sheets obtained by ECAP and subsequent cold and warm rolling are represented in Figs. 2 (a) and (b), respectively. At the same time the original coarse-grained material was cracked upon cold rolling. The present data suggest that the grain refinement to the submicron level of the current hard-to-deform Al-Mg-Sc alloy may provide its enhanced rollability, even at ambient temperature and the alloy can be undergone by extensive deformation without any cracking (see Fig. 2(a)). As it was mentioned above, the possible reason for the premature failure of coarse-grained Al-Mg-Sc alloys with high Mg content is a very inhomogeneous plastic flow, which takes place under cold-to-warm deformation conditions. At that the initial grain size may be of particular importance and localization of plastic deformation may occur much less readily in the fine-grained material [e.g. 10]. Thus the tendency for crack initiation would be decreased with decreasing grain size.

The other important factor that could significantly affect the crack appearance during cold rolling might be the presence of the second phase particles in the material structure. It has been reported in the previous work [6] that before ECAP, the present Al-Mg-Sc alloy contained relatively coarse primarily particles $\text{Al}_6(\text{Fe},\text{Mn})$. These particles were distributed non-uniformly in the original structure and their size was varied from 5 to 30 μm . During cold rolling the latter could cause the stress concentrations followed by a rapid nucleation of cracks in the Al matrix [6]. After ECAP, in contrast, the size of the second phases decreased to the submicrometer scale range and their distribution became more uniform, as these

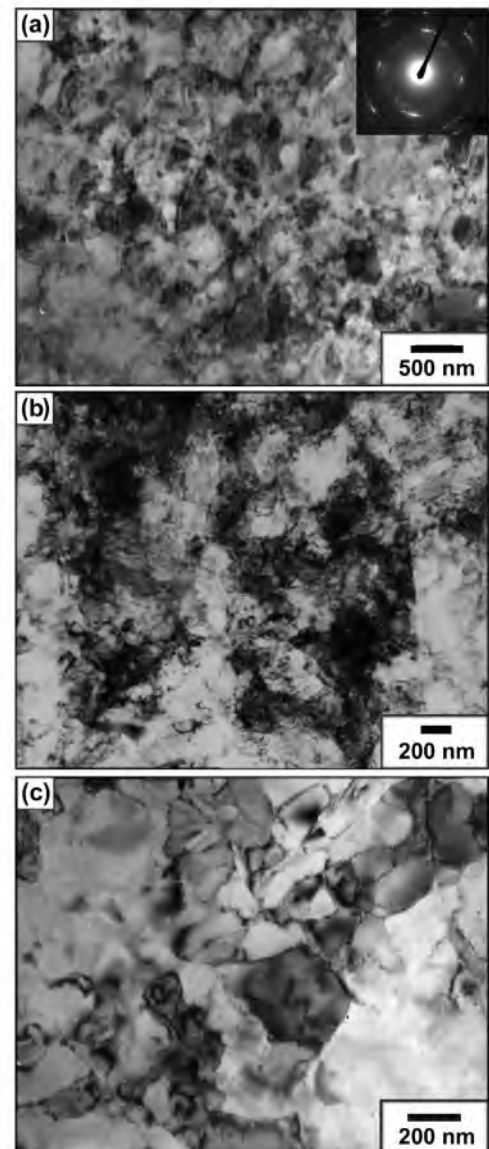


Fig. 3. Typical microstructures of Al-6%Mg-0.3%Sc alloy developed after ECAP and subsequent cold rolling (CR).

phases were profoundly fragmented and/or possibly dissolved during warm-to-hot ECAP. As the result, the harmful effect of the second phases on the crack initiation might become much weaker after ECAP. In the other words, ECAP could provide additional improvement of rollability of the present Al-Mg-Sc alloy due to a fragmentation and/or dissolution of coarse primary particles.

Microstructure after ECAP and subsequent cold rolling. The typical microstructure evolved upon cold rolling with reduction 80% is represented in Fig. 3. It is seen in Fig. 3(a) that the UFG structure evolved during ECAP (Fig. 1(b)-(c)) is replaced by a new heavily deformed microstructure. A detailed micrograph of this microstructure at larger magnification is represented in Fig. 3(b). High-density dislocations arranged in almost equiaxed cellular substructures with the average crystallite size of 0.3 μm can be revealed. The density of lattice dislocations in this structure was $\sim 10^{15}$ - $6 \times 10^{15} \text{ m}^{-2}$. Besides the crystallites with the average size of about 0.2 μm were observed between the areas of such cellular structure. Those were evolved in colonies and practically free of lattice dislocations (Fig. 3 (c)). The volume fraction of such structural component was, however, small and did not exceed 10-20%. The elongated spot character of the selected-area diffraction patterns in Fig. 3 (a) suggested relatively large misorientations of strain-induced boundaries developed. Thus, the deformed microstructure develops during cold rolling of the present alloy in an apparently heterogeneous manner. It is safe to assume that this microstructure may be similar to that reported for some cold-deformed cubic metals with original coarse-grained structure, in which the deformation cell substructures and the new ultrafine grains may be simultaneously present as two structural components at moderate-to-large strains [e.g. 11, 12].

Effect of isothermal warm rolling on the microstructure developed by ECAP. It has been found in the present work that isothermal rolling carried out at the temperature of previous ECAP (i.e. at $T=325^\circ\text{C}$) resulted mainly in increased uniformity of UFG structure. It should be noted in Fig. 1(b) that the microstructure obtained by ECAP is characterized by unrecrystallized grains that exist stably even after larger strains ($\epsilon \sim 12$ -16) [7, 8]. Actually, it is well known that under ECAP conditions, new grains do not usually consume the whole material volume and some unrecrystallized areas, i.e. relatively coarse unrefined fragments of original grains are frequently present in the deformed microstructure [e.g. 8, 10]. These frequently survive to large strains and sustain no significant deformation, since any deformation applied to the material may be mainly relaxed by the surrounded fine-grained matrix developed during ECAP.

Figs. 4(a) and (b) represent the typical optical and TEM microstructures of the present Al-Mg-Sc alloy that were obtained after ECAP and subsequent isothermal rolling with reduction 85%. It is clearly seen in Fig. 4(a) that, in contrast to ECAP, the coarse unrecrystallized grains that were present in the UFG matrix are frequently pancaked upon warm rolling and, after a large reduction, their opposite boundaries come in touch with each other thereby leaving more equiaxed and finer grain structure. Such mechanism of grain refinement may be considered as a kind of geometric dynamic recrystallization [10]. Accordingly, the volume fraction of new fine grains increases from 86pct. after ECAP to 95% after rolling. Thus, the change in deformation mode from ECAP to rolling may result in additional grain structure refinement through extensive transformation of unrecrystallized areas, which are deformed by rolling in accordance to the macroscopic plastic strain of the whole sample. At the same time, it is interesting to note in Fig. 4 that the other parameters of UFG structure, e.g. the size and shape of submicrocrystalline (sub)grains that are evolved during ECAP remain virtually unchanged during subsequent rolling. This may suggest that

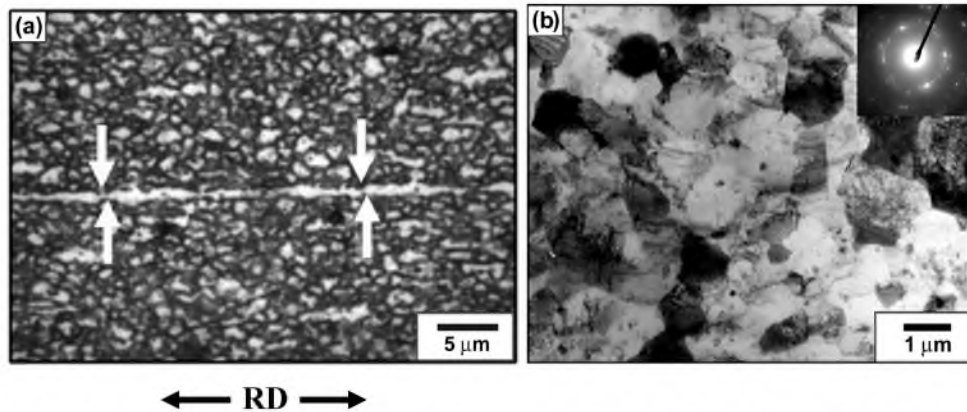


Fig. 4. Typical optical (a) and TEM (b) microstructures developed in Al-6%Mg-0.3%Sc alloy during ECAP and subsequent isothermal warm rolling (WR). RD is the rolling direction.

the plastic deformation in the UFG regions may mainly occur by grain boundary sliding, which may be attributed to the increasing rolling temperature [7, 9].

Mechanical properties. The engineering σ - ϵ curves of the present alloy after ECAP and after ECAP and subsequent cold and warm rolling are represented in Fig. 5. The yield strength (σ_{YS}), ultimate strength (σ_{UTS}) and total elongation until failure (δ) are summarized in Table 1. The same data regarding the original coarse-grained material before ECAP are also shown in Fig. 5 and Table 1 for reference. It is seen that the shape of all curves in Fig.5 is essentially similar. Jerky flow related with the Portevien-Le Chatelier effect [13] takes place. It is interesting to see that extensive strain hardening, $d\sigma/d\epsilon$, associated with high uniform elongation, ϵ_u , is observed after reaching the yield strength, even in cold-rolled UFG material. Note that such mechanical behavior of the present high-strength Al alloy resulting in relatively large values of ϵ_u may contradict the sudden drop in uniform elongation taking place in the pure aluminum, when their grain size is decreased to about 1 μm [14]. It is suggested that in contrast to the pure materials, a strong interaction of the lattice dislocations with Al_3Sc dispersion particles and/or Mg atoms in Al solid solution of the current alloy takes place [5-9] and may in itself enhance work hardening stage that provides a high uniform elongation in spite of largely decreased grain size.

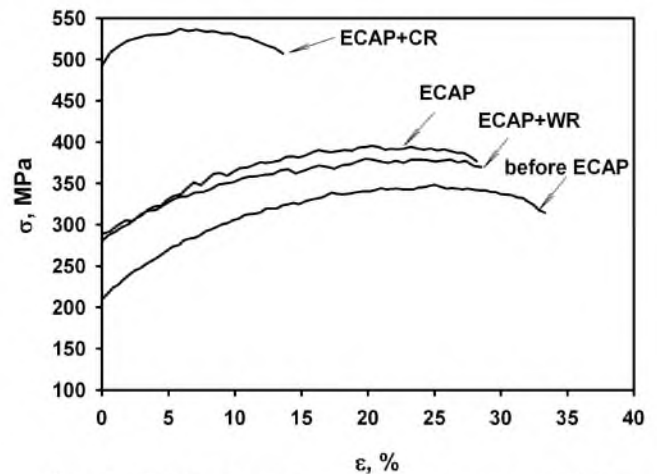


Fig. 5. Typical engineering σ - ϵ curves of Al-6%Mg-0.3%Sc alloy after appropriate thermomechanical treatment.

It is also seen that the formation of UFG structure results in increase in the yield stress from 223 MPa to 285 MPa and ultimate stress from 350 MPa to 389 MPa in comparison with initial hot extruded condition (Table 1). Total elongation slightly decreases from 33% to 29%. Isothermal rolling at a temperature of 325°C to a total strain of ~80-85% following ECAP results in σ_{YS} =290 MPa, σ_{UTS} = 380 MPa, δ =29%. Thus, the mechanical properties after ECAP and ECAP followed by isothermal rolling are roughly similar. In contrast the cold rolling to the same strain results in significant increase of yield stress (495 MPa) and ultimate stress (536 MPa). Total elongation decreased to 13%. This high strength of the ECAP and cold-rolled material may result from the superposition of several factors, i.e. grain refinement and dislocation strengthening of the material hardened by nanoscale dispersoids. Namely, ECAP can provide remarkable grain refinement, while

subsequent cold rolling may introduce heavily deformed substructures with enhanced dislocation density.

Table 1. Mechanical properties of Al-6%Mg-0.3%Sc alloy

Condition	YS (0.2%),(MPa)	UTS,(MPa)	Elongation,(%)
Before ECAP	223±7	350±9	33±3
ECAP	285±5	389±4	29±2
ECAP+WR	290±2	380±3	29±3
ECAP+CR	495±6	536±8	13±2

Summary

Microstructure and mechanical properties of the Al-6%Mg-0.3%Sc alloy were examined upon combination of ECAP at 325°C and subsequent cold- and isothermal warm rolling carried out to high strains. The main results may be summarized as follows.

1. ECAP to total strain 8 results in almost uniform grain structure with the grain size of about 1µm and volume fraction of about 0.86. Such grain refinement to the submicron level enhances rollability of the current hard-to-deform Al-Mg-Sc alloy and provides the achievement of high rolling strains even at ambient temperature.
2. Cold rolling of the alloy with original UFG structure results in a heavily deformed microstructure with high dislocation density. Grain refinement during ECAP and subsequent dislocation strengthening during rolling of the material hardened by nanoscale dispersoids provides yield and ultimate strength of 495 and 536 MPa, respectively.
3. Isothermal warm rolling carried out at the temperature of previous ECAP leads mainly to increase the volume fraction of UFG grains from 86 to 95%, while the other structural characteristics of the material after ECAP remain virtually unchanged. Yield and ultimate strength after warm rolling are 290 and 380 MPa, respectively, and roughly equal to the appropriate properties obtained after ECAP.
4. Extensive strain hardening, associated with high uniform elongation, takes place during tensile test of the present alloy after ECAP and after ECAP and subsequent cold and warm rolling. The material exhibits a relatively high uniform elongation of about 10% even in the very high-strength-state obtained by cold rolling.

Acknowledgments

This work was supported by the International Science and Technology Center under Project no. 2011.

References

- [1] R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov: *Progr. Mater. Sci.* Vol. 45 (2000), p.103.
- [2] R.Z. Valiev, T.G. Langdon: *Progr. Mat. Sci.* Vol. 51 (2006), p. 881.
- [3] H. Akamatsu, T. Fujinami, Z. Horita, T.G. Langdon: *Scr. Mat.* Vol. 44 (2001), p. 759.
- [4] Y.A. Filatov, V.I. Yelagin, V.V. Zacharov: *Mat. Sci. Eng. A* Vol. 280 (2000), p. 97.
- [5] T.G. Nieh, L. M. Hsiung, J. Wadsworth, R. Kaibyshev: *Acta Mat.* Vol. 46 (1998), p. 2789.
- [6] E. Avtokratova, R. Kaibyshev, O. Sitdikov, Y. Watanabe: *Phys. Met. Metall.* Vol. 107 (2009), p. 291.
- [7] F. Musin, R. Kaibyshev, Y. Motohashi, G. Itoh: *Met. Mat. Trans. A* Vol. 35 (2004), p. 2383.

- [8] O. Sitdikov, E. Avtokratova, R. Kaibyshev, T. Sakai, K. Tsuzaki, Y. Watanabe: *Mat. Sci. For.* Vol. 584-586 (2008), p. 481.
- [9] O. Sitdikov, T. Sakai, E. Avtokratova, R. Kaibyshev, K. Tsuzaki, Y. Watanabe: *Acta Mat.* Vol. 56 (2008), p. 821.
- [10] F.J. Humphreys, M. Hatherly: *Recrystallization and Related Annealing Phenomena*. (Elsevier 2004).
- [11] C. Kobayashi, T. Sakai, A. Belyakov, H. Miura: *Phyl. Mag. Lett.* Vol. 87 (2007), p. 751.
- [12] T. Sakai, A. Belyakov, H. Miura: *Met. Mat. Trans. A* Vol. 39 (2008), p. 2206.
- [13] X-M. Cheng, J.G. Morris: *Scr. Mat.* Vol. 43 (2000), p. 651.
- [14] N. Tsuji, Y. Ito, Y. Saito, Y. Minamino: *Scr. Mat.* Vol. 47 (2002), p. 893.