Effect of multiaxial forging on microstructure and mechanical properties of Mg-o.8Ca alloy

2014 IOP Conf. Ser.: Mater. Sci. Eng. 63 012075

(http://iopscience.iop.org/1757-899X/63/1/012075)

Effect of multiaxial forging on microstructure and mechanical properties of Mg-0.8Ca alloy

N Yu Yurchenko[a], N D Stepanov[a], G A Salishchev[a], L L Rokhlin[b], S V Dobatkin[b]

[a]Laboratory of Bulk Nanostructured Materials, Belgorod State University, 85 Pobeda Str., Belgorod, 308015, Russia

[b]A.A. Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences, 49 Leninskii pr., 119991, Moscow, Russia

E-mail: nikitos969@gmail.com

Abstract. It was shown that multiaxial forging with continuous decrease of temperature from 450°C to 250°C turns coarse structure of the Mg-0.8Ca alloy in homogenized state with grain size of several hundreeds µm into fine structure with average grain size of about 2.1 µm. Refinement of structure is accompanied by drastic increase of mechanical properties: tensile yield strength increases from 50 MPa to 193 MPa, ultimate tensile strength increases from 78 to 308 MPa and elongation to fracture increases from 3.0% to 7.2%. The microstructural evolution during multiaxial forging is studied using optical microscopy, scanning electron microscopy and EBSD analysis. The mechanisms responsible for refinement of microstructure are discussed.

Keywords: Magnesium alloy, multiaxial forging, microstructure, mechanical properties.

1. Introduction

Mg and its alloys are promising materials for producing biodegradable implants, due to their excellent biocompatibility [1-3]. However, usage of magnesium alloys is prohibited by some significant disadvantages such as high dissolution rate, low strength and ductility. To tailor the properties, some specific alloy systems for biomedical applications are currently being developed, such as Mg-Ca or Mg-RE (RE= rare earth) [4, 5]. Another approach for improving properties is refinement of structure via thermomechanical treatment. Grain refinement is known to be an effective way of increasing both strength and ductility of most magnesium alloys [6-8]. Application of severe plastic deformation (SPD) results in formation of ultrafine grained (UFG) structure in many metallic materials [9-11]. Microstructure and properties of Mg and many commercial magnesium alloys like AZ31, AZ91, ZK60 after SPD processing were widely studied [12-14]. However, no information is available for effect of SPD on special biomedical Mg alloys. Thus the aim of current study is to study microstructure and mechanical properties of Mg-0.8Ca alloy processed by multiaxial forging (MAF) with continuously decreased temperature. MAF was chosen as it is the one of the few of SPD methods capable of producing relatively large-scale billets [15].

2. Experimental procedures

An initial material used in this study was magnesium alloy Mg-0.8%(wt)Ca. The alloy was produced by resistance furnace melting and cast in a metal mold . The as-cast ingot had approximate dimensions of 40 mm in diameter and 120 mm in length. Samples for forging with dimension of 10 mm in diameter and 14 in length were cut from the ingot using electric discharge machine. Additional large-scale sample with approximate dimensions of 20 mm in diameter and 30 mm in length were produced to examine tensile mechanical properties. All samples were subjected to homogenization annealing at 510°C for 6 hours on air. MAF was performed by sequential "upset-drawing" operation with changing axis of applied deformation force. Instron 300LX machine equiped with radial furnace was used. Each cycle concluded 3 increments of 50-70% compressions. As the result true strain per 1 cycle was ≈ 2.5. Initial strain rate during compression was $2*10^{-3}$ s^{-1}. Stress-strain curves were recorded during each compression. After completing the full cycle dimensions of sample were almost equal to the initial ones. Temperature of forging decreased from 450°C to 250°C with 25°C decreaments per cycle. Thus maximal number of cycles equaled to 9, which corresponded to maximum strain of ≈ 22.5. To study evolution of microstructure, investigations were additionally performed on samples after 1, 3, 5 and 7 passes.

Microstructure was studied by means of optical microscopy (OM) and scanning electron microscopes (SEM) equipped with backscattered electron (BSE) detector, energy dispersive spectrometer (EDS) and electron back-scattering diffraction (EBSD) system. OM observations were performed using Olympus GX 51 microscope. Specimens for the OM observation were prepared by mechanically grinding and polishing the surface of the sample in a diluted OP-S solution and etching it in a solution of 60 ml ethylene glycol, 20 ml acetic acid, 19 ml distilled water and 1 ml nitric acid. BSE characterization and EDS analysis was performed using Quanta 200 3D SEM. EBSD maps were obtained utilizing FEI Nova NanoSEM 450. Prior to analysis samples were carefully prepared by mechanically grinding and polishing the surface of the sample in a diluted OP-S solution.

Tensile test was performed with an INSTRON 5882 testing machine at room temperature and initial strain rate of $1*10^{-3}s^{-1}$. Gauge dimensions of the specimens for tensile test were 16 mm in length, 3 mm in width and 1.5 mm in thickness.

2. Results

Optical photography of microstructure in as-cast state of magnesium alloy Mg-0.8Ca is presented in fig.1a. The microstructure consists from α-Mg matrix and second phase appearing dark on optical micrograph and bright on SEM image (fig 2a.). From EDS analysis data the phase is identified as Mg_2Ca. The average grain size of the α-Mg matrix is about 65 μm, however, some coarse grains with size about 200 μm are also observed (fig.1a). Mg_2Ca phase is found both as an individual equiaxial or elongated particles and as continuous cellular network. The average size of individual particles is 2.5 μm and the average thickness of cell walls is 1.1 μm. One could also note segregations around the Mg_2Ca particles appearing grey on the OM image. These segregations are completely eliminated after homogenization annealing (fig. 1b). Previously continuous network of Mg_2Ca phase is mostly transformed into chains of reasonably equiaxed particles (fig 2b) similar to individual particles which underwent sight coarsening and spheroidization upon annealing. The average size of the particles could be estimated as of 2.3 μm. Some very fine particles of Mg_2Ca phase with average size of 0.4 μm could also be found after homogenization. The average grain size in the homogenized alloy is roughly estimated as of several hundred microns.

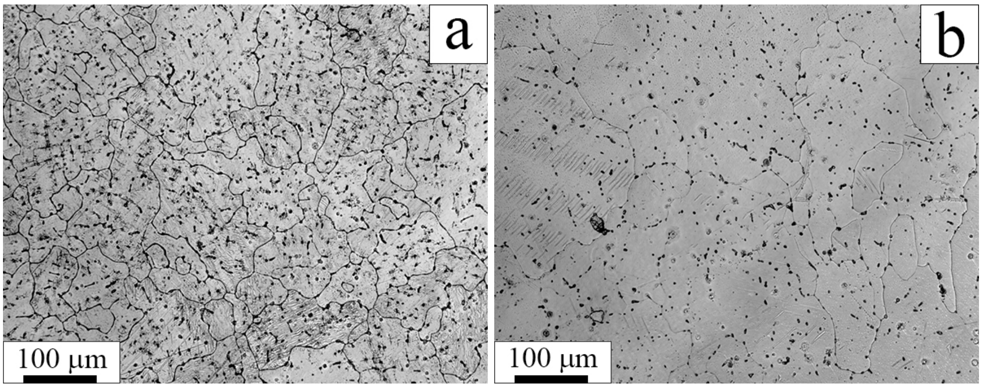

Fig.1. Microstructure of magnesium alloy Mg-0.8Ca: a – as-cast state (OM); b – after homogenization (OM)

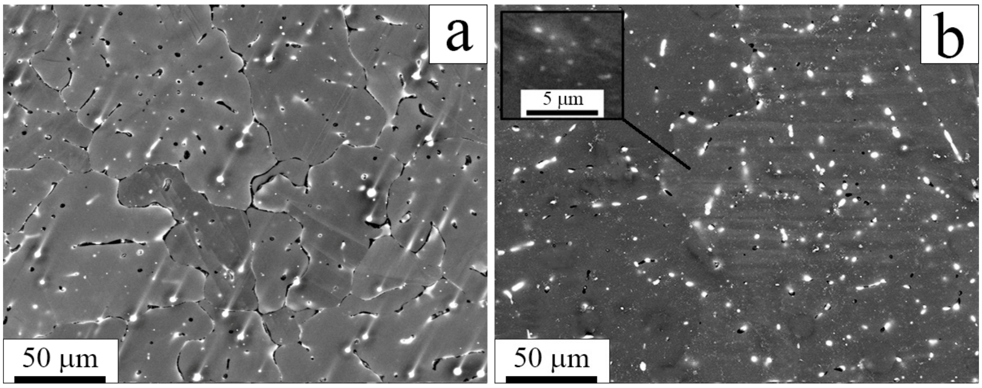

Fig.2. Microstructure of magnesium alloy Mg-0.8Ca: a – as-cast state (a – BSE -SEM); b – after homogenization (b – BSE -SEM).

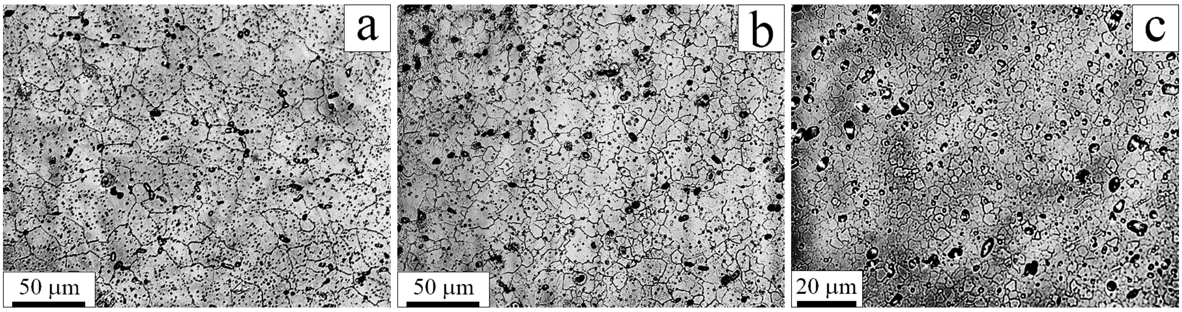

Fig.3. Microstructure of magnesium alloy Mg-0.8Ca after MAF (optical microscopy): a – 1 cycle; b – 5 cycles; c – 9 cycles.

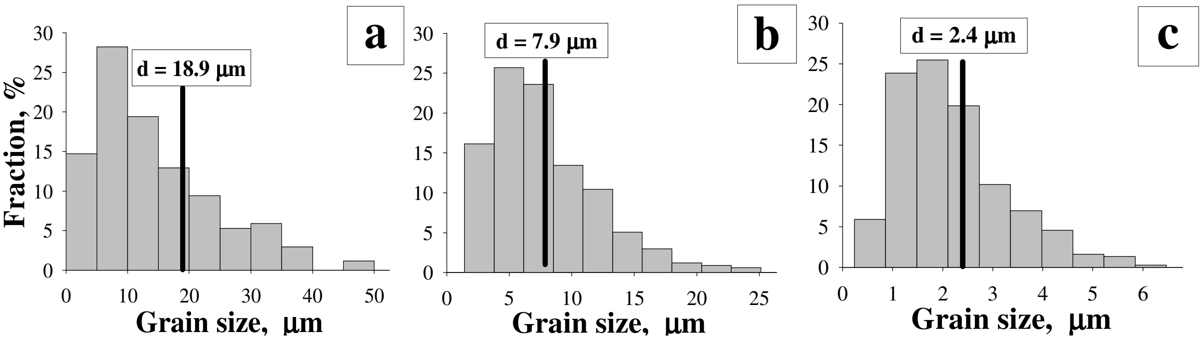

Fig.4. Histograms of grain size distribution of magnesium alloy Mg-0.8Ca after MAF: a – 1 cycle; b - 5 cycles; c – 9 cycles.

Much finer recrystallized microstructure is formed already after first cycle of MAF performed at the temperature of 450°C (fig 3a). However, the structure is not homogeneous and relatively fine grains coexist with much coarser ones. Decreasing deformation temperature down to 350°C (fig 3b) results in decrease of average grain size, but the structure remains inhomogeneous. Further decreasing temperature of forging to 250°C (fig 3c) leads to formation of fine and reasonably homogeneous recrystallized microstructure. The second phase particles, appearing dark on OM images, seem to be unaffected by forging. Grain size distributions are shown on fig. 4. It could be clearly seen that there is no change in character of grain size distribution upon increase of forging cycles from 1 to 9 and corresponding decrease of the processing temperature from 450°C to 250°C. However, the maximum size of grains gradually decreases about 50 µm to about 6 µm and the mean grain size decreases from 18.7 µm to 2.4 µm, again indicating significant grain refinement during MAF.

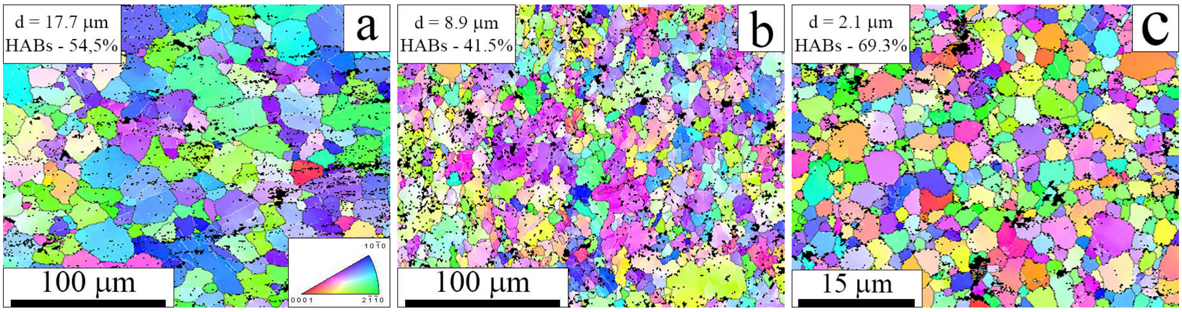

Fig.5. Inverse pole figure (IPF) maps of magnesium alloy Mg-0.8Ca after MAF: a – 1 cycle; b - 5 cycles; c – 9 cycles.

Additional information on microstructure of the Mg-0.8Ca alloy was obtained using EBSD analysis (fig 5). After forging at 450°C somewhat inhomogeneous recrystallized structure with average fraction of high angle boundaries (HABs) of 54.5% was formed (fig. 5a). After 5 passes (forging at 350°C) structure consists mainly of relatively fine grains, however, individual coarse grains are also observed. It's worth to note that these coarse grains are frequently subdivided by low angle boundaries (LABs). The fraction of HABs after 5 passes of MAF is 41.5% (fig 5b). Finally, after forging at 250°C reasonably homogeneous microstructure is formed and fraction of HABs increases to 69.3% (fig. 5c). The grain size determined from EBSD maps is close to those from OM data, as it is shown on fig 6a. With the decrease of temperature and increase of passes number it decreases almost linearly. After 1 cycle of MAF mean grain size was about 17.7 µm, after 5 cycles – 8,9 µm, and after 9 cycles –2,1 µm.

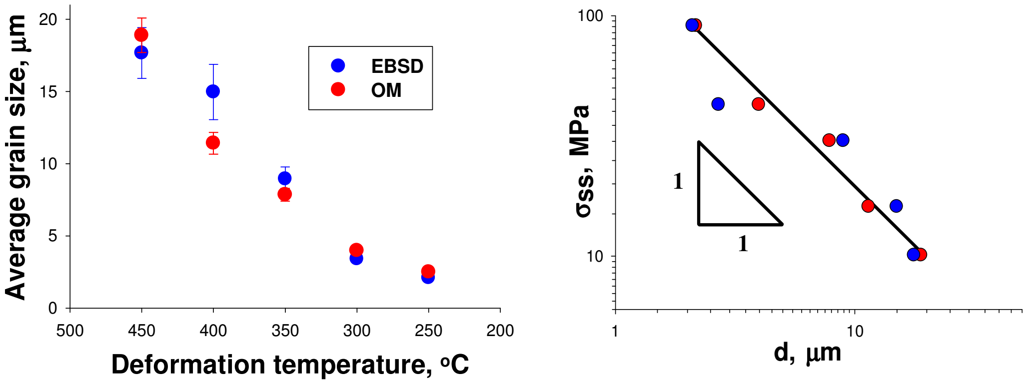

Fig.6. Relationships between processing parameters and grain size determined from OM images (red marks) and EBSD IPF maps (blue marks) of magnesium alloy Mg-0.8Ca during MAF: a – dependence of average grain size on processing temperature and passes number; b – dependence of steady state flow stress on grain size.

Fig. 6b demonstrates the dependence of steady state flow on mean grain size determined both by OM and EBSD. All the experimental points could be approximated with linear dependence with slope equal to 1 with reasonable confidence.

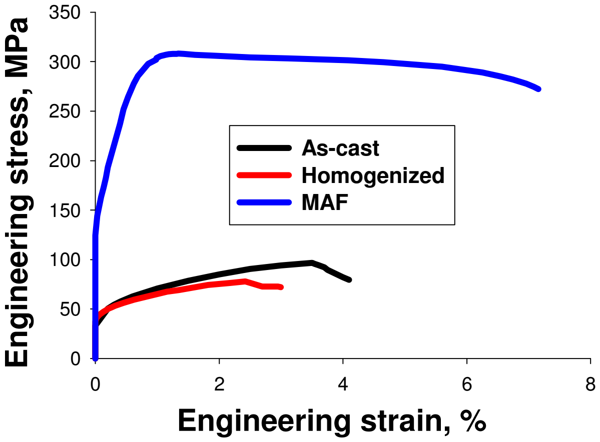

Fig.7. Engineering stress-strain curves after tensile test of magnesium alloy Mg-0.8Ca.

Table 1. Mechanical properties of Mg-0.8Ca.

	σ_{YS}, MPa	σ_{UTS}, MPa	δ, %
As-cast	50.5	96.8	4.1
Homogenized	50.0	77.9	3.0
MAF	193.2	308.2	7.2

Engineering stress-strain curves, obtained during tensile testing of Mg-0.8Ca alloy in different states, are demonstrated on fig. 7. Mechanical properties determined during testing such as yield strength, σ_{YS}, ultimate tensile strength, σ_{UTS}, and elongation to fracture, δ, are summarized in table 1. In as-cast state the alloy has demonstrated low strength – the value of yield strength equals to 50 MPa and value of ultimate tensile strength is of 97 MPa. The ductility of as-cast alloy is also limited – elongation to fracture value is only of 4.1%. Homogenization results in some deterioration of mechanical properties, for example σ_{UTS} decreases to 78 MPa and δ decreases to 3.0%. The σ_{YS} preserves its value of 50 MPa. After multiaxial forging significant improvement of strength is found. Yield strength value increases in about 4 times, to 193.2 MPa, and ultimate tensile strength value increases to 308 MPa. Ductility is also improved and elongation to fracture value increases to 7.2%. Thus one could state that MAF results in substatntial improvement of all mechanical properties of the Mg-0.8Ca alloy.

3. Discussion

Unlike many metallic materials, such as copper or aluminum alloys which could be easily processed by SPD at room temperatures [9], magnesium and its alloys have very low ductility at room temperatures due to limited number of slip systems and thus could be processed only at elevated temperatures when additional slip systems are activated. For example, ECAP processing of Mg alloys is mostly carried out at temperatures of 250-300°C. At such high temperatures, microstructure formation is usually governed by dynamic recrystallization (DRX) [12]. In current work, the Mg-0.8Ca alloy was multiaxialy forged at continuously decreased from 450°C to 250°C temperature. High temperature of the beginning of deformation was chosen due to two features of initial microstructure. At first, the alloy after casting and homogenization annealing had very coarse grains (up to several hundred μm). At second, despite the fact the continuous network of Mg_2Ca phase was broken upon annealing treatment, particles still formed prolonged chains. Both of these factors are expected to limit the plasticity and may promote inhomogeneous deformation. That is why, to ensure sufficient ductility and obtain homogeneous structure, forging was started at 450°C.

Our results demonstrate that one cycle of forging at 450°C is enough to produce reasonably homogeneous recrystallized structure in Mg-0.8Ca alloy with average grain size of 17.7 μm. With decrease of forging temperature, grain size gradually decreases and reaches value of 2.1 μm after forging at 250°C. It is worth to note that one could anticipate changes in processes governing formation of microstructure when the temperature decreases from 450°C to 250°C. However, the grain size distributions (fig. 4) and overall appearance of microstructure (fig. 3 and 5) indicate that the only considerable change in microstructure is decreased grain size. The dependence of steady state flow stress, σ_{ss}, on average grain size provides additional prove that microstructural development during forging is governed by one process, as it follows the same linear dependence in the whole temperature range. Usually, during hot working grain size and flow stress are via the empirical equation $\sigma_{ss}=kd^{-n}$, where k is a constant and n is grain size exponent. In case of multiaxial forging of Mg-0.8Ca alloy, n=1. This fact implies that discontinuous dynamic recrystallization (DDRX) occurs during forging at temperatures from 450°C to 250°C. DDRX is expected to happen in magnesium alloys at high temperatures such as 450°C, but at low temperatures of 250°C one could anticipate continuous dynamic recrystallization (CDRX) [16]. Both phase composition of the alloy (i.e. presence of particles) and extensive structural refinement occurred during MAF could extend temperature range of DDRX, but this matter requires additional studies.

Table 2. Mechanical properties of some UFG Mg alloys.

Alloy	grain size, μm	σ_{YS}, MPa	σ_{UTS}, MPa	δ, %
Mg-0.8Ca (this work)	2.1	193.2	308.2	7.2
AZ31 [12]	1.2	~280	~350	~17
Mg-3Al-0.4Mn [17]	~2.2	~160	~230	55
Mg–5.25 wt.% Zn–0.6 wt.% Ca [18]	~3.9	178	276	25.9
AZ31 [19]	2.2	144	254	60
AZ31 [20]	2.2	-	~175	3
AM60 [21]	~2	~180	~290	~18

Heavy structural refinement had very pronounced effect on mechanical properties of Mg-0.8Ca alloy. It caused significant strengthening and some enhancement of ductility in comparison with both as-cast and homogenized states. Both effects are commonly observed in magnesium alloys after SPD processing [12, 17-21]. Strengthening is usually attributed to grain refinement, and increase of ductility is often connected with formation of beneficial texture alongside with fine grain size. Comparison between mechanical properties of studied Mg-0.8Ca alloys and some other UFG magnesium alloys (corresponding properties are given in table 2), demonstrates that the studied alloy is somewhat stronger than most of the alloys with similar grain size, but has significantly lower ductility. Both higher strength and lower ductility of Mg-0.8Ca alloy could be attributed to presence of Mg_2Ca particles: fine particles, appeared after homogenization, provide strengthening, and coarse particles could limit tensile ductility. Although texture which was not studied in these work also plays important role in determining mechanical properties of the Mg-0.8Ca alloy.

Conclusions

1) Multiaxial forging with continuous decrease of the temperature was successfully used to refine coarse initial microstructure of Mg-0.8Ca alloy. After 9 cycles of forging with final cycle performed at 250°C fine-grained structure with average grain size of 2.1 μm and fraction of HABs of 69.3% was formed. Microstructural development during MAF was found to be governed by DDRX.
2) Microstructure refinement was accompanied by substantial increase of mechanical properties of the Mg-0.8Ca alloy. The yield stress increased in about 4 times in comparison with as-cast and homogenized states, to 193.2 MPa and ultimate tensile strength increased in more than 3 times, to 308.2 MPa. Tensile ductility also increased in about 2 times, elongation to failure after forging was of 7.2%.

References

[1] Virtanen S 2012 *Biomed. App.* **55** pp 101–125
[2] Denkena B et al 2011 *Special Issues on Magnesium Alloys* pp 109–128
[3] Waizy H et al 2013 *J Mater. Sci.* **48** pp 39–50
[4] Li Z et al 2008 *Biomaterials* **29** pp 1329–1344
[5] Li Y et al 2011 *J Mater. Sci.* **46** pp 365–371
[6] Barnett M R et al 2007 *Mater. Sci. Forum* **558-559** pp 433–440
[7] Tong L B et al 2009 *Mater. Sci. and Eng.* **A523** pp 289–294
[8] Miura H et al 2011 *Mater. Sci. and Eng.* **A528** pp 6981–6992
[9] Valiev R Z, Langdon T G 2006 *Prog. in Mater. Sci.* **51** pp 881–981
[10] Zhilyaev A P, Langdon T G 2008 *Prog. in Mater. Sci.* **53** pp 893–979
[11] Saito Y et al 1999 *Acta mater.* **47** no. 2 pp 579–583
[12] Sakai T and Miura H 2011 *Magnesium Alloys - Design, Processing and Properties* ed. Frank Czerwinski (Croatia) chapter 10 pp 219-244
[13] Jorge A del Valle et al 2003 *Mater. Trans.* **44** no. 12 pp 2625–2630
[14] Orlov D et al 2011 *Acta Mater* **59** pp 375–385
[15] Zherebtsov S V, Salishchev G A et al 2004 *Scripta Mater.* **51** pp 1147–1151
[16] Galiev A, Kaibyshev R And Gottstein G 2001 *Acta Mater.* **49** pp 1199-1207
[17] Biswas S and Suwas S 2012 *Scripta Mater.* **66** pp 89–92
[18] Tong L B et al 2010 *Mater. Sci. and Eng.* **A527** pp 4250–4256
[19] Kim W J and Jeong H T 2005 Mater. Trans. **46** no. 2 pp 251–258
[20] Utsunomiya H et al 2009 *J Phys.: Conf. Series* **165** 012011
[21] Kulyasova O et al 2009 *Mater. Sci. and Eng.* **A 503** pp 176–180