Effect of Heat Treatment on the Portevin-Le Chatelier Effect of Al-2.5%Mg Alloy

A. Chatterjee, A. Sarkar, N. Gayathri, P. Mukherjee and P. Barat

Abstract—An experimental study is presented on the effect of microstructural change on the Portevin-Le Chatelier effect behaviour of Al-2.5%Mg alloy. Tensile tests are performed on the as received and heat treated (at 400 ℃ for 16 hours) samples for a wide range of strain rates. The serrations observed in the stress-time curve are investigated from statistical analysis point of view. Microstructures of the samples are characterized by optical metallography and X-ray diffraction. It is found that the excess vacancy generated due to heat treatment leads to decrease in the strain rate sensitivity and the increase in the number of stress drop occurrences per unit time during the PLC effect. The microstructural parameters like domain size, dislocation density have no appreciable effect on the PLC effect as far as the statistical behavior of the serrations is considered.

Keywords—Dynamic strain ageing, Heat treatment, Portevin-Le Chatelier effect

I. INTRODUCTION

THE Portevin-Le Chatelier (PLC) effect, also known as jerky flow, denotes a plastic instability, which is related to the discontinuous plastic flow and plastic strain inhomogeneities. It has been observed in many dilute metallic solid solutions including both interstitial [1] and substitutional [2]. The effect is manifested as serrations in the stress-strain (time) curve. The general consensus explains the origin of the PLC effect as Dynamic Strain Aging (DSA) - the dynamic interaction between the moving dislocation and the diffusing solute atoms [3-8]. The mobile dislocations which are the carrier of the plastic strain move jerkily between the obstacles provided by the other defects. Mobile solute atoms diffuse in the stress field generated by the mobile dislocations and tend to cluster at dislocations. Clustering leads to an enhancement of the apparent lattice resistance to dislocation motion. Clustering is assumed to occur either by lattice diffusion from the lattice to the arrested mobile dislocation, or by pipe diffusion, from the solute cluster on the forest dislocation along the core of the mobile dislocation [9-12].

5*xxx* series Al-Mg alloys have been the model system for studying the Portevin-Le Chatelier effect for a long time [13-17]. These aluminum alloys with nominal percentage of magnesium exhibit the PLC effect at room temperature for a wide range of strain rates. Several works have been done on the serrated behavior with regard to temperature, strain rate, solute atoms etc. However, only few studies have been reported on the effect of microstructure on the serration behavior of the PLC effect [18,19]. In this work, an attempt has been made to characterize the effect of microstructural changes caused by heat treatment of the sample on the PLC effect in Al-2.5% Mg alloy.

II. EXPERIMENTAL METHODS AND ANALYSIS

A. Tensile test

Tensile specimens with dimensions (25 mm × 5 mm × 2.3 mm) have been prepared from an Al-2.5%Mg alloy sheet. Some of the samples are heat treated at 400℃ for 16 hours and then water quenched. Tensile experiments are carried out on the as received and the heat treated samples in an INSTRON (model 4482) machine at room temperature for a wide range of strain rates, starting from $2.0\times10^{-5}s^{-1}$ to $2.0\times10^{-3}s^{-1}$. The machine is computer controlled and the stress data are recorded at an interval of 0.05 seconds.

B. Optical metallography

Samples for optical micrograph were cut from the as-received and the heat-treated samples.

A. Chatterjee is with Variable Energy Cyclotron Centre, Kolkata - 700064, India (corresponding author phone: +91 33 23182406; fax: +91 33 23346871; e-mail: arnomitra@vecc.gov.in).

A. Sarkar is with Bhabha Atomic Research Centre, Mumbai – 400085, India. (e-mail: apusarkar@gmail.com).

N. Gayathri is with Variable Energy Cyclotron Centre, Kolkata - 700064, India (e-mail: gayathri@vecc.gov.in).

P. Mukherjee is with Variable Energy Cyclotron Centre, Kolkata - 700064, India (e-mail: paramita@vecc.gov.in).

P. Barat is with Variable Energy Cyclotron Centre, Kolkata - 700064, India (e-mail: pbarat@vecc.gov.in).

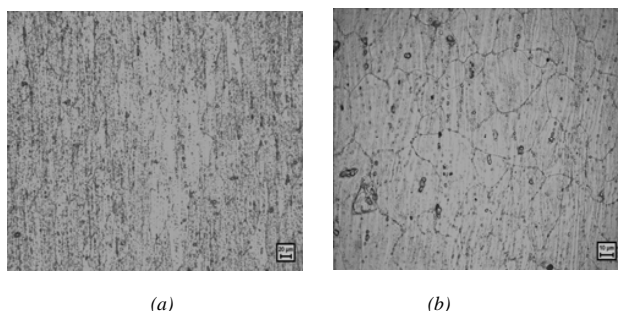(a)   (b)

Fig. 1 Optical micrograph of (a) as received and (b) heat treated sample.

They were cold mounted and mechanically polished. The polishing agents for the intermediate and final polishing processes were diamond suspensions with particle sizes of 9

and 3 μm and alumina. The solution used for etching was a mixture of 1 parts hydrofluoric acid and 99 parts distilled water. Fig. 1(a) and 1(b) show the optical micrographs of the as received and the heat treated samples respectively.

C. X-Ray diffraction

XRD is a powerful tool to characterize the microstructure of the polycrystalline samples. It gives the microstructure of the sample in a statistical manner. XRD profiles for the as received and the heat treated samples have been recorded by Bruker D8 Advance diffractometer using *CuK$_\alpha$* radiation. The range of 2θ was from 35° to 85° and a step scan of 0.02° was used. The time per step was 4 seconds. Fig. 2(a) and 2(b) show the XRD profile of the as received and the heat treated samples respectively. It is evident from these figures that the texture of the sample has changed due to the heat treatment.
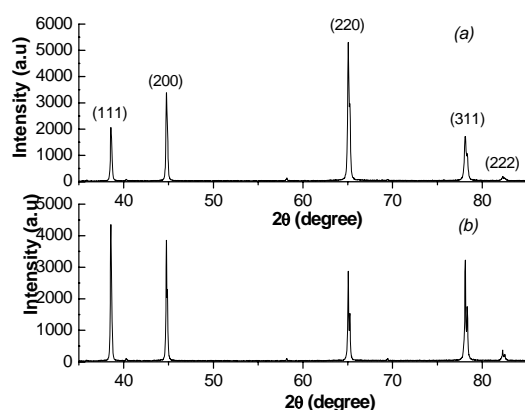

Fig. 2 XRD profile of (a) as received and (b) heat treated sample

Microstructural characterization from X-ray diffraction line profile analysis has become a very popular technique in recent years. Various methods have been developed to determine the microstructural parameters like domain size, microstrain, dislocation density from the broadened XRD peaks [20, 21]. We have used the variance method developed by Groma [22, 23] and the Integral Breadth method [24] to characterize the microstructure of the samples.

D. Studies on the PLC effect

Fig. 3 shows the typical stress-strain curves of the as received and the heat treated samples deformed at a strain rate of 3.8×10^{-4} s^{-1}. It is seen that the ductility of the sample has changed drastically due to the heat treatment. However, here we are interested in the effect of heat treatment on the PLC effect. To quantify the changes in the behaviour of PLC effect we concentrate on the statistical features of the serrations. We have carried out the following studies on the stress-time data recorded during the PLC effect of the as received and heat treated samples.

Negative strain rate sensitivity is considered as the essential criterion for the PLC effect. The strain rate sensitivity parameter, *m*, was evaluated from the constant strain rate tests

using the equation $m = \dfrac{\log[\sigma_1 / \sigma_2]}{\log[\dot{\varepsilon}_1 / \dot{\varepsilon}_2]}$ [25], where σ_1 and σ_2 represent the flow stress at the current strain, measured in tests performed with strain rates $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$, respectively.
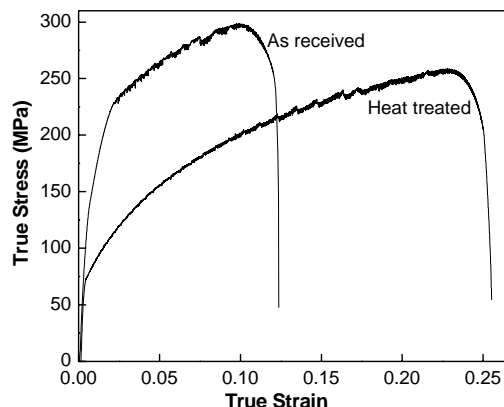

Fig. 3: True stress vs. True strain curve for as received and heat treated sample deformed at a strain rate of 3.8×10^{-4} s^{-1}.

TABLE I
STRAIN RATE SENSITIVITY PARAMETER FOR THE AS RECEIVED AND THE HEAT TREATED SAMPLES. $\dot{\varepsilon}_1$ =3.7×10^{-4} s^{-1} $\dot{\varepsilon}_2$ =6.5×10^{-4} s^{-1}.

Sample	Strain	Strain rate sensitivity parameter
	0.05	-0.042
As received	0.06	-0.022
	0.08	-0.014
	0.09	-0.024
	0.05	-0.126
Heat treated	0.10	-0.084
	0.15	-0.120
	0.20	-0.119

For two chosen strain rates we have computed *m* at various strain values. Typical values of the *m* for the as received and the heat treated samples are listed in Table I.

Number of stress drop per unit time

The number of stress drops (nsd) per unit time gives the measure of the activity of the PLC dynamics. The variation of the average nsd per second with strain for both the as received and the heat treated samples are shown in Fig. 4.
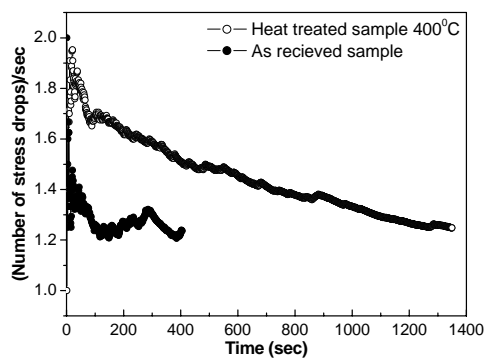
Fig. 4: Variation of average number of stress drops per second for as received and heat treated sample.

Reloading time

Another parameter which is often studied in the PLC effect literature is the time during the stress increase. This time is called the reloading time and it corresponds to the time for which the band waits at the obstacles prior to its release due to thermal activation. We have calculated the reloading time corresponding to each stress drop in the stress-time series data.
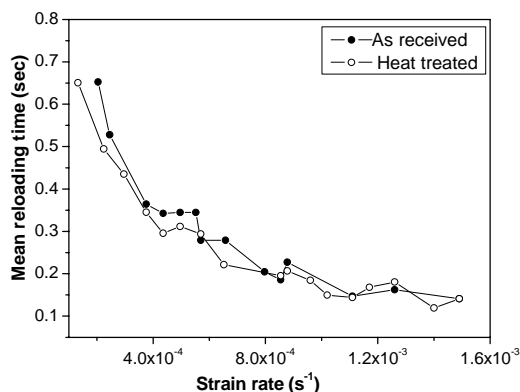

Fig. 5: Variation of average reloading time for as received and heat treated sample.

Fig. 5 shows the variation of the mean reloading with the strain rate.

Stress drop magnitude

Magnitude of stress drops during the PLC effect is considered as one of the important parameter to characterize the PLC effect. The different types of serrations, now well known in the literature of the PLC effect, were first identified from the study of the distribution of the stress drop magnitude [26, 27]. Fig. 6(a) and 6(b) show the variation of the average stress drop magnitude with the strain rate for the as received and the heat treated samples in double-logarithmic scale. It is seen that the average stress drop magnitude decreases with the strain rate for both as received and heat treated samples. Fig. 7(a) and 7(b) show the cumulative distribution of the stress drop magnitude for the as received and the heat treated

samples deformed at three representative strain rates. The inset of the figures shows the corresponding frequency distribution plot. The distribution of nsd and reloading time are also computed from the stress-time series data.
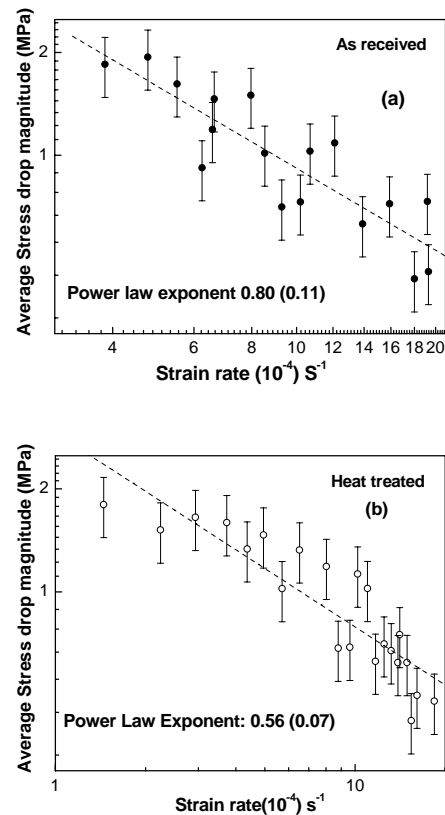

Fig. 6: Variation of average stress drop magnitude for (a) as received and (b) heat treated sample.

III. RESULTS AND DISCUSSIONS

From the optical micrographs of Fig. 1 it is evident that the grain structure has changed substantially after the heat treatment. XRD line profile analysis gives quantitative details of the microstructure of the sample. The microstructural parameters obtained from X-ray diffraction line profile analysis of the as received and the heat treated samples (Table II and Table III) revealed that the domain (sub-grain) size has increased due to heat treatment, while the dislocation density and the microstrain have decreased on heat treatment.

TABLE II
MICROSTRUCTURAL PARAMETERS OBTAINED FROM THE VARIANCE METHOD ANALYSIS OF THE (111) PEAK OF THE AS RECEIVED AND THE HEAT TREATED SAMPLES

Sample	Domain size (Å)	Dislocation density (10^{14}) (m^{-2})
As received	390 (±35)	8.19 (±0.26)
Heat treated	461 (±43)	6.23 (±0.19)

TABLE III
MICROSTRUCTURAL PARAMETERS OBTAINED FROM THE
INTEGRAL BREDTH ANALYSIS OF THE AS RECEIVED AND THE
HEAT TREATED SAMPLES

Peak	As received		Heat treated	
	Domain size (Å)	Microstrain (10^{-4})	Domain size (Å)	Microstrain (10^{-4})
(111)	616	9.06	707	5.50
(200)	760	9.13	962	7.25
(220)	615	9.10	693	4.39

Moreover, the visual inspection of the XRD profiles of both the samples indicates that the texture of the sample has also changed due to heat treatment.
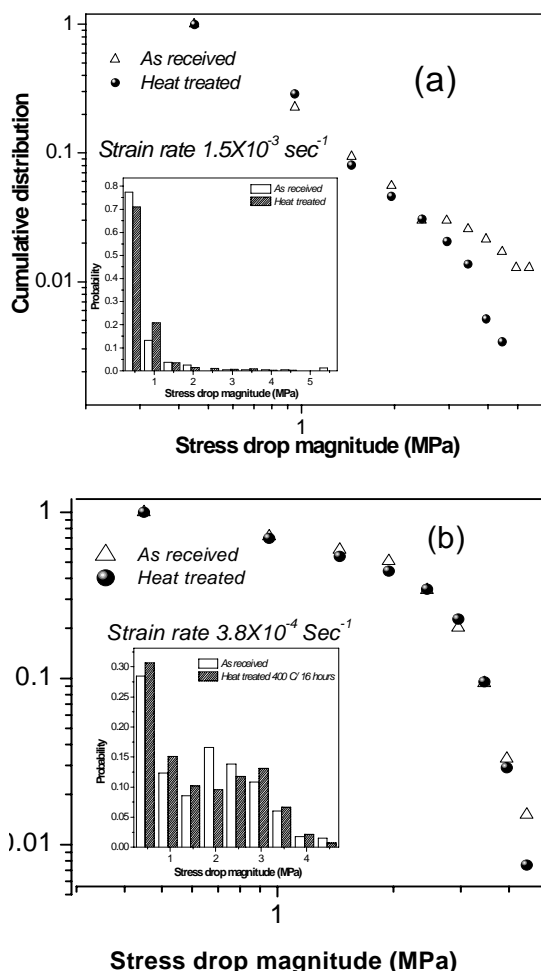

Fig. 7: Probability distribution of stress drop magnitude for strain rate (a) 3.8×10^{-4} s^{-1} (b) 1.5×10^{-3} s^{-1}.

Al-Mg alloy is a substitutional alloy. The atomic radius of Mg atom is 12% larger than the Al atom. The diffusion of solute Mg atom is the primary ingredient of the DSA and hence the PLC effect in this alloy. Diffusion of Mg in Al-Mg is vacancy assisted. Therefore, the excess vacancies present in the heat treated sample are expected to have influence on the

PLC serrations. From Table I it is seen that the m parameter is negative for both the samples. Moreover, the value of m is more negative in case of the heat treated sample. This result corroborates with the earlier observation of Picu et. al. [25] in an Al-4.5%Mg alloy. It has been established in previous studies that grain size has no significant effect on the SRS [28]. The dislocation density is also expected to have no effect on the SRS [29]. Thus the pronounced reduction of the SRS in the heat treated sample may be attributed to the enhanced mobility of the solute in the presence of excess vacancies.

The variation of the nsd per second in Fig. 4 shows that the nsd per second is more in the heat treated sample as compared to the as received sample. This signifies the frequent occurrence of the locking-unlocking phenomenon of the band in the heat treated sample. This may be attributed to the fact that the presence of excess vacancy in the heat treated sample increases the diffusion coefficient of Mg atom and makes the locking mechanism faster. This increases the number of stress drop occurrences.

On the other hand, the distribution of stress drop magnitude (fig.7) at a particular strain rate appear to be almost similar for the as received and the heat treated samples. This holds true for the distribution of nsd and reloading time also. The plot of variation of the average reloading time with strain rate in Fig. 5 are also similar for both the as received and the heat treated samples.

It is now well established that during the PLC effect deformation in the material localizes in a narrow band. The deformation of the material in this case is entirely governed by the movement of the band of dislocations. In recent years, there have been some highly sophisticated efforts to study the kinematical properties of the PLC bands [30-32]. These studies estimated various band parameters like band width, band velocity, inclination angle of the band with the tensile axis etc. All studies indicated that the PLC bands are not confined to a particular slip plane but are wide and extended to few grains. This may be attributed to the reason behind the similarity of the probability distributions of nsd per second, stress drop magnitude, reloading time for a particular strain rate experiment of the as received and the heat treated samples. The observed serrations in the stress-time curve are manifestation of the jerky band movement in the mesoscopic scale of the material. Thus the serrations are the outcome of the effect averaged over a few grains. Hence, the changes in grain size, dislocation density and texture have no appreciable effect in the statistical behaviour of the PLC effect.

In one of their studies, Klose et. al. [33] have shown that the average stress drop magnitude decreases in a power law fashion with strain rate at a particular temperature, i.e., the variation of the average stress drop magnitude ($\Delta\sigma$) with strain rate (ε) can be expressed by the following relation

$$\Delta\sigma = \dot{\varepsilon}^{\alpha}$$

The power law exponent α has been shown to depend on the diffusion mechanism of the solute atoms and the dislocation pile-up configuration [33]. From Fig. 6(a) and 6(b) it is seen that the $\Delta\sigma$ show a power law dependence with ε for both the as received and the heat treated samples. The values of the exponent α obtained from the slope of the linear fits

are 0.80 ($\pm$0.11) and 0.56 ($\pm$0.0.07) for as received and heat treated samples respectively. The values of α are markedly different from that obtained by Klose et. al. for Al-1.5%Mg alloy deformed at room temperature. This may be due to the difference in the dislocation pile-up configuration in these two materials. However, the change in the value of α in case of the heat treated sample from the as received sample is definitely due to the change in the diffusion mechanism of solute atom. This again is related to the presence of excess vacancy in the heat treated sample.

IV. CONCLUSIONS

In conclusion, we have studied the effect of heat treatment on the behaviour of the PLC effect in Al-2.5%Mg alloy. The heat treatment of the sample has generated excess vacancy in the lattice and has changed the microstructure and the texture of the sample. Analysis of the stress time series data revealed that the excess vacancy in the heat treated sample has changed the SRS and the number of stress drop occurrence per unit time in the heat treated sample. The microstructural parameters like domain size, dislocation density and microstrain do not have observable influence on the PLC effect.

REFERENCES

[1] A. Sarkar, A. Chatterjee, P. Barat and P. Mukherjee, "Comparative study of the Portevin-Le Chatelier effect in interstitial and substitutional alloy," Mat. Sci. Eng. A, vol. 459, pp. 361-365, June 2007.

[2] P. Barat, A. Sarkar, P. Mukherjee, S.K. Bandyopadhyay, " Scaling behavior of the Portevin-Le Chatelier effect in Al-2.5%Mg alloy," Phy. Rev. Lett., vol. 94, pp. 055502.1-055502.4, Feb. 2005.

[3] A.H. Cottrell, *Dislocations and plastic flow in crystals,* London, Oxford University Press, 1953.

[4] P.G. McCormick, "A model for the Portevin-Le Chatelier effect in the substitutional alloys," Acta Metall., vol. 20, pp. 351-354, March, 1972.

[5] J.D. Baird, *The Inhomogeneity of Plastic Deformation*, American Society of Metals, OH, 1973.

[6] A. Van den Beukel, "Theory of the effect of dynamic strain aging on mechanical properties," Phy. Status Sol (a), vol. 30, pp. 197-206, Feb. 1975.

[7] A. Van den Beukel, U.F. Kocks, "The strain dependence of static and dynamic strain aging," Acta Metall., vol. 30, pp. 1027-1034, May 1982.

[8] J. Schliph, "On the kinetics of static and dynamic strain aging," Scripta Metall. Mater., vol. 31, pp. 909-914, Oct. 1994.

[9] R.C. Picu, "A mechanism for the negative strain rate sensitivity of dilute solute solutions," Acta Mater., vol. 52, pp. 3447-3458, July 2004.

[10] H. Jiang, Q. Zhang, X. Chen, Z. Chen, Z. Jiang, X. Wu, J. Fan, "Three types of Portevin -Le Chatelier effect: Experiment and modeling," Acta Mater., vol. 55, pp. 2219-2228, April 2007.

[11] A.W. Sleeswyk, " Slow strain hardening of ingot iron," Acta Metall., vol. 6, pp. 598-603, Sep. 1958.

[12] R.A. Mulford, U.F. Kocks, "New observations on the mechanisms of dynamic strain aging and of jerky flow," Acta Metall., vol. 27, pp. 1125-1134, July 1979.

[13] F. Chmelik, F.B. Klose, H. Dierke, J. Sachl, H. Neuhauser, P. Lukac, "Investigating the Portevin- Le Chatelier effect in strain rate and stress rate controlled tests by the acoustic emission and laser extensometry techniques," Mat. Sci. Eng. A, vol. 462, pp. 53-60, July 2007.

[14] J.M. Robinson and M.P. Shaw, "The influence of specimen geometry on the Portevin- Le Chatelier effect in an Al-Mg alloy," Mat. Sci. Eng. A, vol. 159, pp. 159-165, Dec. 1992.

[15] K. Chihab, Y. estrin, L.P. Kubin and J. Vergnol, " The kinetics of the Portevin-Le Chatelier bands in in an Al-5at%MG alloy," Scripta Metall., vol. 21, pp. 203-208, Feb. 1987.

[16] J. Balik, P. Lukac and L.P. Kubin, " Inverse critical strains for Jerky flow in Al-Mg alloys," Scripta Mater., vol. 42, pp. 465-471, Feb. 2000.

[17] M. Abbadi,P. Hahner and A. Zeghloul, "On the characteristics of the Portevin-Le Chatelier bands in aluminum alloy 5182 under stress controlled and strain controlled tensile testing," Mat. Sci. Eng. A, vol. 337, pp. 194-201, Nov. 2002.

[18] B.J. Roopchand and J.G. Morris, "The effect of cold work on the strain rate sensitivity of 5086 aluminum alloy," Scripta Metall., vol. 7, pp. 839-845, Aug. 1973.

[19] Wei Wen and J.G. Morris, "The effect of cold rolling and annealing on the serrated yielding phenomenon of AA5182 aluminum alloy," Mat. Sci. Eng. A, vol. 373, pp.204-216, May 2004.

[20] Defect and microstructure analysis by diffraction, R.L. Synder, J. Fiala and H.J. Bunge, Ed. New York, Oxford University Press, 1999.

[21] Diffraction analysis of the microstructure of materials, vol. 68, E.J. Mittemeijer and P. Scardi Ed. Berlin, Springer-Verlag, 2004.

[22] I. Groma, "X-ray line broadening due to an inhomogeneous dislocation distribution," Phy. Rev. B, vol. 57, pp. 7535-7542, April 1998.

[23] A. Borbely, I. Groma, "Variance method for the evaluation of the particle size and dislocation density from x-ray Bragg peaks," App. Phy. Lett., vol. 79, pp. 1772-1774, Sep. 2001.

[24] Th. H.de Keijser J. I.Langford E. J.Mittemeijer A. B. P.Vogels, "Use of the Voigt function in a single-line method for the analysis of X-ray diffraction line broadening," J. Appl. Cryst., vol. 15, pp. 308-314, June 1982.

[25] R.C. Picu, G. Vincze, J.J. Gracio and F. Barlat, "Effect of solute distribution on the strain rate sensitivity of solute solutions," Scripta Mater., vol. 54, pp. 71-75, Jan 2006.

[26] M.A. Lebyodkin, Y. Brechet, Y. Estrin and L.P. Kubin, "Statistics of the Catastrophic Slip Events in the Portevin–Le Châtelier Effect," Phy. Rev. Lett., vol. 74, pp. 4758-4761, June 1995.

[27] M. Lebyodkin, Y. Brechet, Y. Estrin and L.P. Kubin, "Statistical behavior and strain localization pattern in the Portevin-Le Chatelier effect," Acta Mater., vol. 44, pp. 4531-4541, Nov. 1996.

[28] J. May, H.W. Hoppel and M. Goken, "Strain rate sensitivity of ultrafine-grained aluminum processed by severe plastic deformation," Scripta Mater., vol. 53, pp. 189-194, July 2005.

[29] L.P. Kubin and Y. Estrin, "Evolution of the dislocation densities and the critical conditions for the Portevin-Le Chatelier effect," Acta Metall. Mater., vol. 38, pp. 697-708, May 1990.

[30] R. Shabadi, S. Kumar, H.J. Roven and E.S. Dwarakadasa, "Characterization of the PLC band parameters using laser speckle technique," Mat. Sci. Eng. A, vol. 364, pp. 140-150, Jan. 2004.

[31] H. Louche, P. Vacher and R. Arrieux, "Thermal observations associated with the Portevin-Le Chatelier effect in an Al-Mg alloy," vol. 404, pp. 188-194, Sep. 2005.

[32] Q. Zhang, Z. Jiang, H. Jiang, Z. Chen and X. Wu, "On the propagation and pulsation of Portevin-Le Chatelier deformation bands: An experimental study with digital speckle pattern metrology," Int. J. Plasticity, vol. 21, pp. 2150-2173, Nov. 2005.

[33] F. B. Klose, A. Ziegenbein, F. Hagemann, H. Neuhäuser, P. Hähner, M. Abbadi, A. Zeghloul, "Analysis of Portevin-Le Chatelier serrations of type B in Al-Mg," Mat. Sci. Eng. A, vol. 369, pp. 76-81, March 2004.