Microstructural characterization of the Portevin–Le Chatelier band in an Al-Mg alloy by X-ray diffraction line profile analysis

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Al–2.5% Mg alloy exhibits the Portevin–Le Chatelier (PLC) effect at room temperature for a wide range of strain rates. Tensile test has been carried out on a flat Al–2.5% Mg alloy sample at a strain rate of 3.7×10^{-6} s^{-1}. The strain rate was chosen so that the type C PLC band appears in the sample. X-ray diffraction profile has been recorded from the gauge length portion of the deformed sample to investigate the microstructure of the PLC band. Analysis revealed that the dislocation density is much higher within the band compared to the undeformed sample even at small strain. The PLC band in this alloy possesses an equal fraction of screw and edge dislocations. © *2010 International Centre for Diffraction Data.* [DOI: 10.1154/1.3478567]

Key words: Portevin–Le Chatelier effect, dislocation, X-ray diffraction, tension test, Al alloy

I. INTRODUCTION

Plastic deformation of materials exhibits many spectacular features and has engaged numerous researchers for a long time in the study of deformation characteristics of materials. The Portevin–Le Chatelier (PLC) effect observed in plastic deformation of many metallic alloys of technological importance (Halim *et al.*, 2007; Hogg *et al.*, 2007; Banerjee and Naik, 1996) is one of the widely studied metallurgical phenomena. It is a striking example of the complexity of spatiotemporal dynamics, arising from the collective behaviour of dislocations. In uniaxial loading with constant imposed strain rate, the effect manifests itself as a series of serrations (stress drops) in the stress time or strain curve (Kubin *et al.*, 2002). Each stress drop is associated with the nucleation of a band of localized plastic deformation, often designated as the PLC band, which under certain conditions propagates along the sample. The microscopic origin of the PLC effect is the dynamic strain aging (DSA) (Kubin *et al.*, 2002; Rizzi and Hahner, 2004; McCormigk, 1972; van den Beukel, 1975; Kubin and Estrin, 1985) of the material due to the interaction between mobile dislocations and diffusing solute atoms. At the macroscopic scale, this DSA leads to a negative strain rate sensitivity of the flow stress and makes the plastic deformation nonuniform.

In polycrystals three types of PLC effect are traditionally distinguished on the qualitative basis of the spatial arrangement of localized deformation bands and the particular appearance of deformation curves (Kubin *et al.*, 2002; Chihab *et al.*, 1987). Three generic types of serrations, types A, B, and C, occur depending on the imposed strain rate. For sufficiently large strain rate, type A serrations are observed. In this case, the bands are narrow, continuously propagating, and highly correlated. The associated stress drops are small in amplitude. If the strain rate is lowered, type B serrations with relatively larger amplitude occur around the uniform stress strain curve. These serrations correspond to intermittent band propagation. The deformation bands are formed ahead of the previous one in a spatially correlated manner and give rise to regular surface markings. At very low strain rate, bands become static and wide. This type C band nucleates randomly in the sample, leading to large saw-tooth shaped serration in the stress strain curve and random surface markings (Kubin *et al.*, 2002; Chihab *et al.*, 1987).

In recent years there have been some highly sophisticated efforts to observe the PLC band and identify the characteristics of the band. Laser speckle technique, digital speckle interferometry, and infrared pyrometry have been used to study PLC bands (Ranc and Wagner, 2005; Ait-Amokhtar *et al.*, 2008; Shabadi *et al.*, 2004). These studies have provided reasonable understanding of the band features such as bandwidth, its velocity, etc. However, what is still lacking in the study of the PLC effect is the microstructural characterization of the band. In this study, we attempt to characterize the microstructural feature of the PLC band.

Traditionally, two complimentary tools used for studying dislocation patterns are X-ray line profile analysis of diffraction patterns and transmission electron microscopy (TEM). While TEM provides detailed information of dislocation structure, it has some serious drawbacks. The patterns observed in the TEM are not representative of the bulk situation as the method requires thin samples whose dimensions are much smaller than the PLC band size that is to be studied. This leads to stress relaxation due to migration of dislocations to the surfaces. On the other hand, X-ray line profile can, in principle, give information of the bulk dynamics but results are averages over grains and orientations. We have used X-ray diffraction (XRD) line profile analysis to investigate the microstructure of the PLC band.

II. EXPERIMENTAL

Al-Mg alloy is a model system to study the PLC effect. It exhibits the PLC effect at room temperature for a wide range of strain rates. We have carried out tensile test on a flat Al–2.5% Mg alloy sample using an INSTRON (model 4482)

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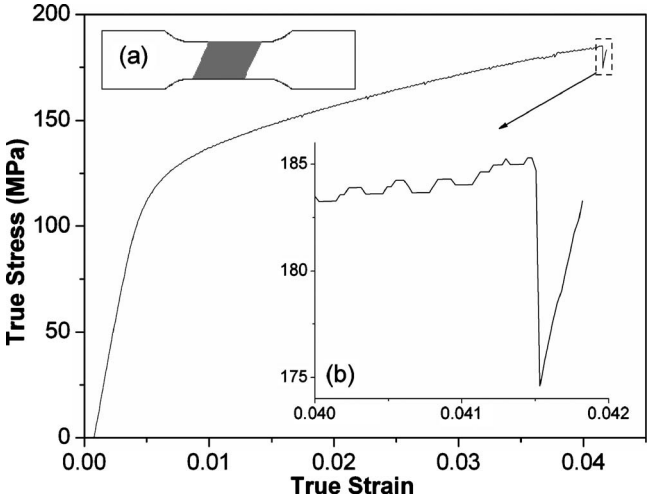

Figure 1. True stress vs true strain curve for Al–2.5% Mg sample deformed at a strain rate of 3.7×10^{-6} s^{-1}. Insets (a) shows the schematic diagram of the tensile specimen depicting the PLC band and (b) shows the magnified view of the large stress drop.

machine. Since our purpose was to investigate the microstructure of the PLC band we did not prepare the tensile sample according to the ASTM specification. The gauge length of the sample was 6 mm. A schematic diagram of the specimen is shown in the inset (a) of Figure 1. The experiment was conducted at a strain rate of 3.7×10^{-6} s^{-1}. The strain rate was chosen so that the type C band appears in the sample (Kubin *et al.*, 2002; Chihab *et al.*, 1987). As reported in the literature the type C band is ~1 to 2 mm wide and is associated with large characteristic load drop. Once we observe a large load drop in the stress strain curve we immediately stop the test and take the sample out of the machine. The representative stress strain curve is shown in Figure 1. By terminating the test in such a fashion we capture a type C band in the specimen. The large load drop is shown in the inset (b) of Figure 1.

XRD profiles of the as received and the deformed samples were recorded with a Bruker D8 Advance powder diffractometer using variable slit arrangement. The irradiated area of the sample was fixed to 6×6 mm^2 to take diffraction only from the gauge length portion. The instrumental broadening was characterized by LaB_6 (SRM 660a) standard sample. Diffraction profiles of the sample have been corrected the instrumental broadening using the Stokes deconvolution method (Stokes, 1948).

III. RESULTS AND DISCUSSION

X-ray diffraction line profile analysis is one of the most powerful methods of determination of microstructure of the deformed materials (Warren, 1969). Several new formalisms of X-ray diffraction line profile analysis have been proposed (Mittemeijer and Scardi, 2004; Snyder *et al.*, 1999) in past few years. Reliable information of intricate details of microstructure of a material is now possible to extract from the XRD line profile. We have adopted few newly developed techniques to characterize the microstructure of the PLC band.

The whole powder pattern fitting technique developed by Dong and Scardi (2000) is a very good method to get the average microstructural information. Figure 2(a) shows the whole powder pattern fit of the deformed Al–2.5% Mg alloy sample. Figure 2(b) shows the Williamson-Hall (WH) plot

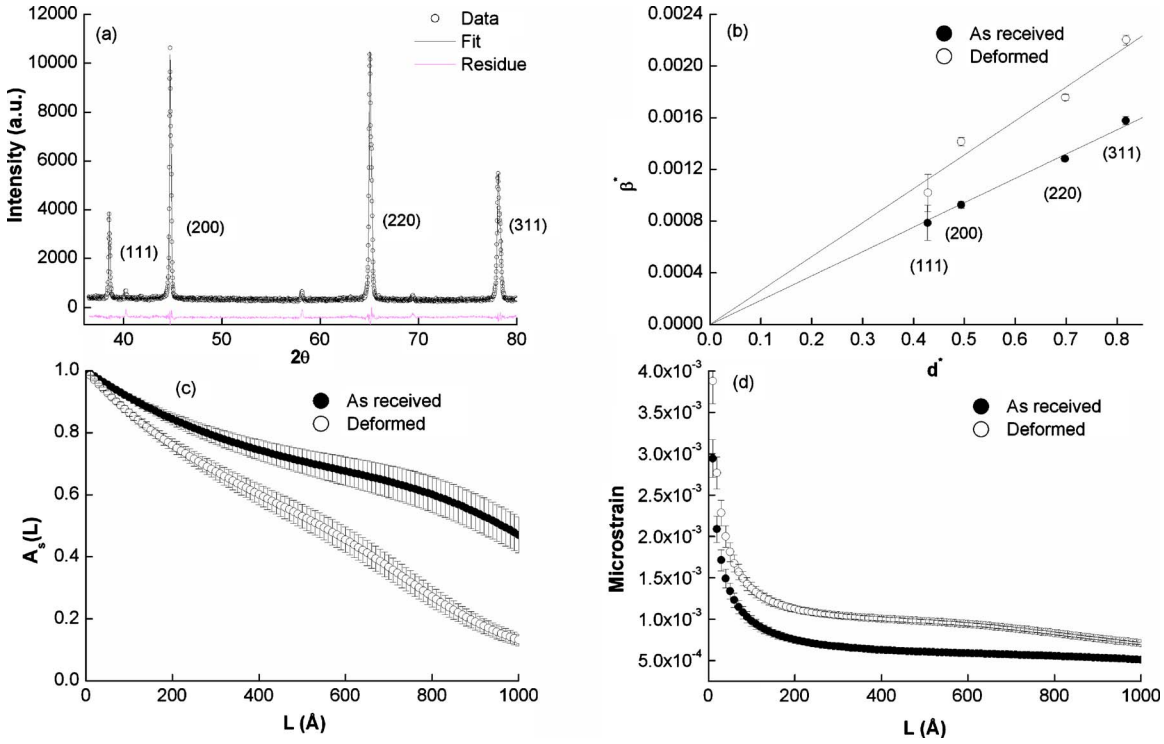

Figure 2. (Color online) (a) Whole powder pattern fit of the XRD pattern of the deformed sample; (b) WH plot of the as received and deformed samples; (c) size Fourier coefficient of WA plot; initial slope of this plot gives the size value; and (d) microstrain plot obtained from WA analysis.

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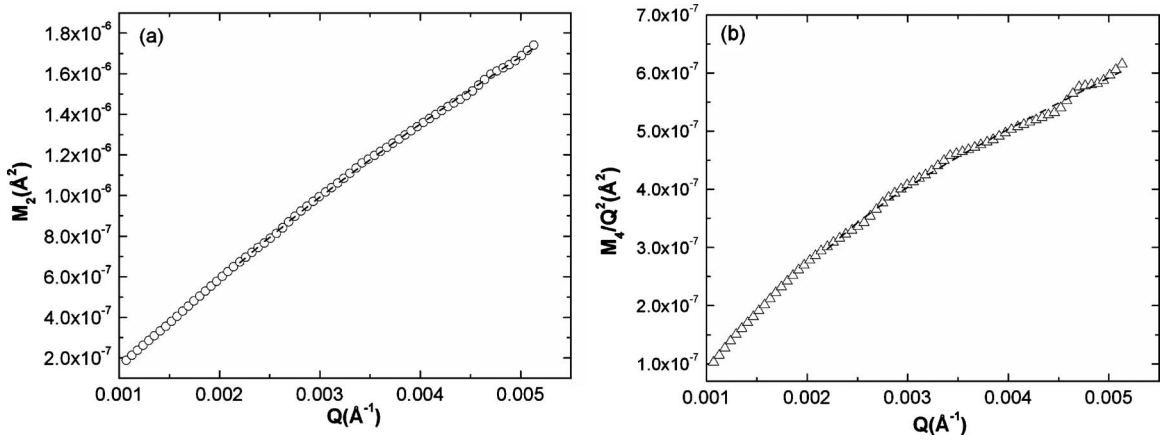

Figure 3. Variation of (a) second and (b) fourth order restricted moments with Q (=2 sin θ/λ) for the (200) peak of the deformed sample. Fitting of asymptotic regime of these plots with equation described by Groma (1998) gives the value of the dislocation density.

(Williamson and Hall, 1953) of the as received and the deformed samples. The WH plots reveal the fact that the size effect in the broadening of the line profile for both the samples is insignificant. The domain size values obtained from the intercept of the WH plots are greater than 100 nm for both the samples. The strain effect to the broadening is quite prominent. The average microstrain values obtained from the fit of the WH plot are $1.88(0.03)\times10^{-3}$ and $2.63(0.08)\times10^{-3}$ for the as received and the deformed samples, respectively. The absence of size effect and high microstrain values can also be verified from the Warren-Averbach (WA) analysis [Figures 2(c) and 2(d)].

We have applied the variance method to estimate the dislocation density in the samples (Groma, 1998; Borbély and Groma, 2001). The variance method is based on the asymptotic behaviour of the second and fourth order restricted moments. Though the mathematical foundation of this theory is similar to the model published earlier (Groma *et al.*, 1988), it is based only on the analytical properties of the displacement field of straight dislocations and no assumption is made on the actual form of the dislocation distribution, and thus it can be employed for the inhomogeneous dislocation distribution. The variance method has been applied to (200) and (220) peaks, which have higher intensities. Figures 3(a) and 3(b) show the plots of the variations of the second and fourth order restricted moments for the (200) peak of the deformed sample. The right level of the background is chosen from the criteria that the domain size values obtained from both the moments are the same (Borbély and Groma, 2001). The estimated dislocation densities for the as received and the deformed samples are $2.0(0.4)\times10^{14}$ and $1.0(0.7)\times10^{15}$ m^{-2}, respectively. However, it is worth mentioning that in the XRD measurement the area illuminated was larger (6×6 mm^2) than the width of the PLC band (~2 mm). Taking into consideration the volume fraction of the PLC band within the X-ray beam, we obtain the value of dislocation density in the PLC band to be $\sim2.5\times10^{15}$ m^{-2}. Thus, the average dislocation density in the PLC band is much higher than the as received sample even at a small strain of $\sim4\%$.

The information about the character of dislocations can be obtained using the modified Williamson-Hall plot (Ungára *et al.*, 1998),

$$\Delta K = \frac{0.9}{D} + \alpha K\sqrt{\bar{C}} + O(K^2\bar{C}), \tag{1}$$

where D is the domain size and α is a constant. $K=2\sin\theta/\lambda$, where θ is the diffraction angle and λ is the wavelength of X-rays. $\Delta K=\cos\theta[\Delta(2\theta)]/\lambda$, where $\Delta(2\theta)$ is the full width at half maximum of the diffraction peak. $\bar{C}$ is the average contrast factor of dislocations depending on the relative orientations of the diffraction vector and the Burger and the line vectors of the dislocations and the elastic constants of the crystal. O stands for the higher order term in $K\sqrt{\bar{C}}$. For cubic polycrystalline material $\bar{C}$ obeys the relation

$$\bar{C} = \bar{C}_{h00}(1 - qH^2), \tag{2}$$

where $\bar{C}_{h00}$ are average dislocation contrast factors for the $h00$ reflections, $H^2=(h^2k^2+h^2l^2+k^2l^2)/(h^2+k^2+l^2)^2$, and q is a parameter which depends on the character of dislocations. Inserting Eq. (2) in Eq. (1) and neglecting the first term (since D is large) we get

$$\frac{\Delta K^2}{K^2} = A(1 - qH^2), \tag{3}$$

where $A=\alpha^2\bar{C}_{h00}$. The value of q can be obtained by plotting $\Delta K^2/K^2$ against H^2. Figure 4 shows these plots for the as received and the deformed samples.

The q parameter values in aluminum for pure screw and edge dislocations in the ⟨110⟩ {111} slip system can be de-

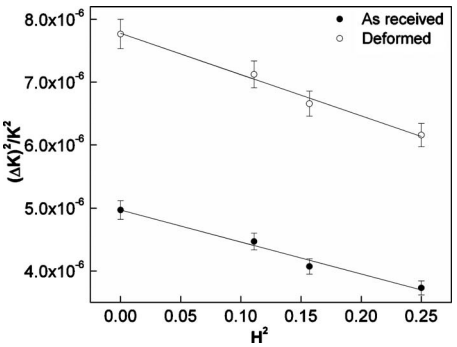

Figure 4. Plot of $\Delta K^2/K^2$ vs H^2 to find q.

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termined by using the detailed numerical calculations and equations of Ungár *et al.* (1999) and the elastic constants of Hearmon (1979). According to this, for pure screw or edge dislocations the values of q are 1.33 and 0.36, respectively. The experimental values (Figure 4) of the q parameters obtained for the present as received and deformed Al–2.5% Mg alloy samples are 1.02(0.08) and 0.84(0.05), respectively. Thus, for the as received sample the value of q is higher than the arithmetic average of pure edge and screw cases, 0.85, which means that the character of dislocations is more screw type. The value of q (0.84) for the deformed sample indicates that the PLC band possesses almost equal fraction of edge and screw dislocations. The increase in the fraction of edge dislocation in the deformed sample may be attributed to the fact that in case of the substitutional alloy Al-Mg, the size mismatch between the host (Al) and the solute (Mg) is high (about 12%) (Picu and Zhang, 2004). This spherically symmetric size misfit effect is the dominant part of the dislocation-solute interaction over the modulus effect (Vannarat *et al.*, 2001). Thus, in the Al-Mg alloy, the solute atoms primarily interact with the edge dislocations. It is well established that the dislocation-solute interaction is the primary reason behind the formation of the PLC band. Hence, more the edge dislocations take part in the formation of PLC band in Al-Mg alloy.

IV. CONCLUSION

Microstructure of the Portevin–Le Chatelier band in Al–2.5% Mg alloy has been investigated by X-ray diffraction line profile analysis. Results of our analyses indicate that

(i) Dislocation density within the PLC band is much higher than that of the undeformed sample even at small strain and
(ii) PLC band in this alloy is comprised of an equal fraction of screw and edge dislocations.

Ait-Amokhtar, H., Fressengeas, C., and Boudrahem, S. (**2008**). "The dynamics of Portevin–Le Chatelier bands in an Al-Mg alloy from infrared thermography," Mater. Sci. Eng., A **488**, 540–546.

Banerjee, S. and Naik, U. M. (**1996**). "Plastic instability in an omega forming Ti-15% Mo alloy," Acta Mater. **44**, 3667–3677.

Borbély, A. and Groma, I. (**2001**). "Variance method for the evaluation of particle size and dislocation density from X-ray Bragg peaks," Appl. Phys. Lett. **79**, 1772–1774.

Chihab, K., Estrin, Y., Kubin, L. P., and Vergnol, J. (**1987**). "The kinetics of the Portevin–Le Chatelier bands in an Al–5 at. % Mg alloy," Scr. Metall. **21**, 203–208.

Dong, Y. H. and Scardi, P. (**2000**). "MARQX: A new program for whole-powder-pattern fitting," J. Appl. Crystallogr. **33**, 184–189.

Groma, I. (**1998**). "X-ray line broadening due to an inhomogeneous dislocation distribution," Phys. Rev. B **57**, 7535–7542.

Groma, I., Ungar, T., and Wilkens, M. (**1988**). "Asymmetric X-ray line broadening of plastically deformed crystals. I. Theory," J. Appl. Crystallogr. **21**, 47–54.

Halim, H., Wilkinson, D. S., and Niewczas, M. (**2007**). "The Portevin–Le Chatelier (PLC) effect and shear band formation in an AA5754 alloy," Acta Mater. **55**, 4151–4160.

Hearmon, R. F. S. (**1979**). "*The Elastic Constants of Crystals and Other Anisotropic Materials*," Landolt-Börnstein Tables, in Landolt-Börnstein Tables, Group III, Pt. 11, edited by K. H. Hellwege and A. M. Hellwege (Springer-Verlag, Berlin), pp. 1–244.

Hogg, S. C., Palmer, I. G., Thomas, L. G., and Grant, P. S. (**2007**). "Processing, microstructure and property aspects of a spraycast Al-Mg-Li-Zr alloy," Acta Mater. **55**, 1885–1894.

Kubin, L. P. and Estrin, Y. (**1985**). "The Portevin–Le Chatelier effect in deformation with constant stress rate," Acta Metall. **33**, 397–407.

Kubin, L. P., Fressengeas, C., and Ananthakrishna, G. (**2002**). "Collective behaviour of dislocations in plasticity," in *Dislocations in Solids*, edited by F. R. N. Nabarro and M. S. Duesbery (Elsevier, Amsterdam), Vol. 11, pp. 101–192.

McCormigk, P. G. (**1972**). "A model for the Portevin–Le Chatelier effect in substitutional alloys," Acta Metall. **20**, 351–354.

Mittemeijer, E. J. and Scardi, P. (**2004**). *Diffraction Analysis of the Microstructure of Materials* (Springer, Berlin).

Picu, R. C. and Zhang, D. (**2004**). "Atomistic study of pipe diffusion in Al-Mg alloys," Acta Mater. **52**, 161–171.

Ranc, N. and Wagner, D. (**2005**). "Some aspects of Portevin–Le Chatelier plastic instabilities investigated by infrared pyrometry," Mater. Sci. Eng., A **394**, 87–95.

Rizzi, E. and Hahner, P. (**2004**). "On the Portevin–Le Chatelier effect: Theoretical modeling- and numerical results," Int. J. Plast. **20**, 121–165.

Shabadi, R., Kumar, S., Roven, H. J., and Dwarakadasa, E. S. (**2004**). "Characterisation of PLC band parameters using laser speckle technique," Mater. Sci. Eng., A **364**, 140–150.

Snyder, R. L., Fiala, J., and Bunge, H. J. (**1999**). *Defect and Microstructure Analysis by Diffraction* (Oxford University Press, Oxford).

Stokes, A. R. (**1948**). "A numerical Fourier-analysis method for the correction of widths and shapes of lines on x-ray powder photographs," Proc. Phys. Soc. London **61**, 382–391.

Ungár, T., Dragomir, I., Révész, A., and Borbély, A. (**1999**). "The contrast factors of dislocations in cubic crystals: The dislocation model of strain anisotropy in practice," J. Appl. Crystallogr. **32**, 992–1002.

Ungára, T., Ott, S., Sanders, P. G., Borbely, A., and Weertman, J. R. (**1998**). "Dislocations, grain size and planar faults in nanostructured copper determined by high resolution X-ray diffraction and a new procedure of peak profile analysis," Acta Mater. **46**, 3693–3699.

van den Beukel, A. (**1975**). "Theory of the effect of dynamic strain aging on mechanical properties," Phys. Status Solidi A **30**, 197–206.

Vannarat, S., Sluiter, H. F. M., and Kawazoe, Y. (**2001**). "First-principles study of solute-dislocation interaction in aluminum-rich alloys," Phys. Rev. B **64**, 224203(1-8).

Warren, B. E. (**1969**). *X-Ray Diffraction* (Addison-Wesley, Reading, MA).

Williamson, G. K. and Hall, W. H. (**1953**). "X-ray line broadening from filed aluminium and wolfram," Acta Metall. **1**, 22–31.