Effect of equal channel angular pressing on the Portevin–Le Chatelier effect in an Al3Mg alloy

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Plastic deformation of alloys is often accompanied with discontinuous flow known as the Portevin–Le Chatelier (PLC) instability. Recent studies of mechanical behavior of alloys subjected to severe plastic deformation (SPD) revealed different forms this phenomenon can take as a result of grain refinement. In the present work, it is reported that SPD may effectively suppress the macroscopic stress serrations characteristic of the PLC effect. At the same time, small-scale stress fluctuations can still be resolved on the deformation curves. To investigate their nature, stress vs time curves for an Al3Mg alloy processed by equal channel angular pressing (ECAP) are compared with those obtained for the initial coarse-grained (CG) material, using statistical and multifractal approaches to multi-scale analysis of time series. An interpretation of the results in terms of the concurrency of phenomena of self-organized criticality and synchronization in the dislocation system is proposed.

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1. Introduction

Grain refinement caused by SPD allows obtaining ultra-fine grained (UFG) materials which possess exceptional mechanical properties and have a great application potential [1]. At the same time, they usually display a low work-hardening capacity [2], which leads to an early loss of stability of uniform plastic deformation and, consequently, strain localization. It is thus not surprising that the influence of SPD on the PLC effect in metallic alloys has recently attracted attention of researchers (e.g., [3–7]). The PLC effect, or jerky flow, manifests itself as repetitive stress serrations associated with strain localization in plastic deformation bands [8]. It is generally believed to be controlled by the mechanism of dynamic strain aging (DSA) of dislocations – a term referring to their interaction with solute atoms during arrest times at localized obstacles [9]. As will be illustrated in Section 3, several specific types of such behavior are usually distinguished when either the applied strain rate or temperature is varied, regarding the temporal patterns of stress serrations and the respective spatial patterns of localized deformation bands [10].

The available literature data on plastic instability in UFG materials, mainly dealing with investigations of the serrated character of deformation curves, do not reveal a unique trend for the effect of the extreme grain size reduction on the unstable plastic flow [3–7]. Although most observations indicate that the strain interval where the PLC effect occurs is usually reduced in UFG materials, both an increase and a decrease in the magnitude of stress serrations were observed for materials processed by SPD of various kinds. In addition, substantial changes in their temporal patterns were often detected. However, a direct comparison of these behaviors is complicated because various authors report data for different deformation conditions.

In the present work, unstable deformation curves of a model Al3Mg alloy are studied in a wide strain-rate range of $2 \times 10^{-5}$–$7 \times 10^{-3}\text{s}^{-1}$ which covers the main types of behaviors associated with the PLC effect at ambient temperature. The deformation curves for the material in the initial CG state are compared with those of the same material processed by ECAP. A specific feature of the approach used is the application of the statistical and multifractal methods of analysis of time series to quantify and compare the temporal patterns of stress serrations. Indeed, these and similar methods have been successfully used in the literature to discriminate between the different types of behavior of the PLC effect in CG alloys [11–19].
2. Experiment and data processing

2.1. Experimental procedure

Cold-rolled 10 mm thick sheet of an Al–3% Mg alloy was used to cut both flat tensile specimens for testing CG material and billets with a 10 × 10 mm² section for ECAP treatment. The dog-bone shaped flat specimens had a gage part 25 × 6.8 × 2.2 mm³ in size. Both kinds of samples were subjected to solution treatment consisting in annealing in air for 2 h at 400 °C, resulting in an isotropic grain structure with approximately equiaxed grains with an average size in a range of about 30–100 μm. ECAP was performed according to route Bc [20], using a die with the channel angle of 90° and an installation described in Ref. [21]. It involved two steps: the billets were passed through the die four times at 6.7–100 °C and then four times at 200 °C. In both cases, the temperature–resistant molybdenum disulfide lubricant was applied. Flat tensile specimens of a slightly smaller size (22 × 5.15 × 2.2 mm³) than the specimens of CG material were then machined from the billet.

Room temperature tensile tests were carried out at a constant crosshead velocity corresponding to the following values of the applied strain rate $\dot{\varepsilon}_0$ for the initial specimen length: $2 \times 10^{-5}$ s⁻¹; $6.7 \times 10^{-5}$ s⁻¹; $2 \times 10^{-4}$ s⁻¹; $6 \times 10^{-4}$ s⁻¹; $1 \times 10^{-3}$ s⁻¹; $7 \times 10^{-3}$ s⁻¹. The combined stiffness of the “machine-specimen” system was approximately $10^7$ N/m. The load cell sensitivity corresponded to the stress resolution near 0.001 MPa for the specimens with the cross-section given above. The sampling time for recording the stress–time curves was chosen in a range from 2 ms for the fastest tests to 500 ms for the slowest ones. The strain value was measured using a clip-on extensometer with a 12.5 mm distance between edges.

The microstructure of the UFG material was investigated by transmission electron microscopy (TEM) in a Philips CM200 electron microscope at an acceleration voltage of 200 kV. Specimens for TEM were cut from ECAP billets perpendicular to the direction of pressing (plane X), then mechanically polished on emery papers down to the thickness of 200 μm. Thin foils were finally electrochemically polished using a Struers Tenupol 5 double-jet polishing unit at −30 °C in the solution of 30% HNO₃ and methanol.

2.2. Analysis of deformation curves

The statistical analysis of the magnitudes of stress serrations was performed in time intervals demonstrating unchanged characteristic shapes of serrations and stationary statistical behavior. The latter was verified by varying the length of the time interval and repeating the analysis. The time series for the multifractal analysis were obtained from the stress–time curves $\sigma(t)$ corrected by subtracting the average curves reflecting the stress evolution due to strain hardening (or softening). The latter were calculated using either a moving average procedure or a polynomial approximation. As suggested in Ref. [13], the multifractal analysis was applied to the time series obtained by taking the absolute value of the numerical approximation $\psi_j$ of the derivative of the corrected stress–time curve, $|d\sigma/dt|$. The multifractal procedure is briefly explained below (see Refs. [13–15] for more detail). The method can be illustrated by studying scaling behavior of the partition functions $S_q(\delta t) = \sum_{i=1}^N |\mu_i(\delta t)|^q$, where a grid of $N$ time steps $\delta t$ is applied to cover the time interval, $\mu_i(\delta t)$ is the so-called local measure given by the normalized integral intensity of the $\psi_j$ “signal” over $\delta t$, $\mu_i(\delta t) = \sum_{j=1}^{\delta t} \psi_{i,j} / \sum_{j=1}^{\delta M} \psi_{j}$, $m$ is the number of data points within $\delta t$, $M$ stands for their total number within the time interval analyzed, and $q$ is a real number. A multifractal signal is characterized by a property of self-similarity which results in a scaling relationship $S_q(\delta t) \propto \delta t^{D(q)–1}$ when $\delta t$ tends to zero, where $D(q)$ is called the generalized fractal dimension [22]. In practice, such scaling is searched for in a significant $\delta t$ interval. This feature allows discriminating self-similar time series from both random and periodic signals, which correspond to uniform behavior for a $\delta t$-range above the period characterizing the signal variations and, consequently, render a trivial scaling law $D(q) \equiv 1$.

3. Experimental results and discussion

3.1. Microstructure of UFG material

Fig. 1(a) presents the typical microstructure of the material after 8 passes of ECAP. In most areas the refined microstructure consisting of bands of elongated fine subgrains/grains of the average width and length of 150–200 nm and 500–700 nm, respectively, was observed.

![Fig. 1](image_url)

**Fig. 1.** The microstructure of Al3Mg processed by ECAP: (a) bands of subgrains/grains, and (b) detail of fragmented grains.
Hence, ECAP processing resulted in grain refinement by roughly two orders of magnitude as compared to the initial grain size range (see Section 2.1). Newly formed fragmented grains were often found in these bands. One example of these grains is shown in Fig. 1(b). Detailed tilting experiments and the analysis of diffraction patterns revealed that these grains contain very low density of dislocations. Extinction contours indicate the presence of large internal stresses in the vicinity of grain boundaries. Almost no banded contrast and the high magnitude of the misorientation angles observed confirm that these boundaries are in a non-equilibrium condition. The occurrence of such non-equilibrium boundaries in materials that underwent severe plastic deformation has been reported repeatedly [1,23,24].

3.2. Overall deformation behavior

Fig. 2 compares examples of deformation curves for four strain rates, obtained before and after ECAP. One example is shown for each εc value. The curves recorded at the same strain rate for other CG samples are quite close to the presented ones. In contrast, the UFG samples only show a perfect reproducibility in terms of the flow stress but exhibit a significant variation in the elongation: the failure strain as small as about 2–3% was observed for some of them. In general, the elongation to failure for UFG specimens is at least 5 times smaller than in the CG case. Furthermore, their ultimate tensile strength indicating the onset of necking is reached very quickly, typically after 1.5–3% strain. By contrast, their maximum flow stress is about twice as high as that of CG specimens. It should be noted, however, that the proportionality limit is attained approximately at the same stress for both kinds of samples, around 50 MPa, so that there is substantial plasticity during the initial steep stress rise in UFG samples.

Before going to the description of stress serrations, it is of interest to present such quantity as the strain-rate sensitivity (SRS) of the flow stress, which is one of the central elements of DSA models of the PLC effect [9]. The deformation curves of CG specimens tend to lie below the curves measured at lower strain rates. (Note that the bias of 10 MPa introduced between the deformation curves in Fig. 2(a) exaggerates the differences but correctly describes this relationship.) This indicates negative strain-rate sensitivity of stress – a property considered as a necessary condition for the onset of the PLC instability. As seen in Fig. 2(b), strain softening sets in, and this occurs the earlier the higher the strain rate is. One may think SRS is negative in this case, too, but this is contravened by the fact that the initial portions of the curves practically coincide. This observation bears witness to a SRS value around zero, corresponding, in a first approximation, to the boundary of stability of plastic flow from the viewpoint of DSA models.

3.3. Temporal patterns of stress fluctuations on multiple scales

The temporal patterns of stress evolution observed in the case of CG materials are similar to those published previously for similar alloys [10–19]. These patterns present special features at multiple scales. Among these, the macroscopic serrations illustrated in Fig. 2(a) and associated with the PLC effect, were studied most extensively [10–18]. Curves 1, 2, and 4 illustrate, respectively, type C, type B, and type A behavior of the PLC effect (cf. Ref. [10]). Curve 3 shows an A to B transition due to work hardening of the material during the test. The range of the magnitude of the serrations is about 10 MPa at 1;3 εfl–10–5 s−1 and decreases down to 2 MPa at 1;4 εfl=7×10−3 s−1. It has been established in literature that the stress serrations are associated with nucleation of deformation bands across the specimen, at an angle about 50–60° to the tensile axis [10]. Type C behavior corresponds to virtually uncorrelated occurrence of deformation bands along the specimen length, each stress drop usually corresponding to the occurrence of one PLC band. Type B pattern corresponds to strongly correlated sequences of bands usually described as relay-race band propagation. Finally, type A behavior is associated with quasicontinuous propagation of bands resulting in plateaus on deformation curves, superimposed with irregular stress fluctuations.

Other aspects of behavior can be seen before the critical strain εcr for the onset of the PLC instability is reached. The magnitude of εcr is particularly important at low strain rates (cf. curve 1). In this case, by increasing the magnification of the curve between the yield point and the critical strain it is possible to observe smaller stress drops (~1 MPa) preceding the onset of the PLC effect, as shown in Fig. 3(a). Such stress drops were for a long time considered as sporadic fluctuations due to the specimen surface imperfections [25]. However, recent investigations testified that they might also be precursors of the PLC instability, when the conditions of a strong synchronization of dislocations necessary to generate macroscopic stress drops are not attained yet [26]. It is noteworthy that stress fluctuations in a similar size range can also be found during the reloading phase between the deep stress drops.

Finally, stress oscillations illustrated in the inset in Fig. 3(a) are also observable before εcr. Their magnitude is about 0.1–0.2 MPa at 1;5 εfl=2×10−8 s−1, but it decreases with an increase in εfl, finally becoming indiscernible at the highest strain rate. The first discussion
of this scale of plasticity in aging alloys was presented in Ref. [19], which related such stress oscillations to concurrent processes of wave-like strain localization and intermittent local plastic activity.

Fig. 3 demonstrates that small stress drops accompany the initial deformation stage of UFG material, too. Compared with the respective observations on CG samples, the stress drops in the UFG case display the same kinetics and similar magnitude, provided the latter is scaled by a factor of approximately two. Similarly to the above case, stress drops are relatively frequent at the low strain rate, but become quite rare at higher strain rates. This similitude is a particularly surprising result; a further important observation in this work is that in contrast to the case of CG material, these small serrations are followed by macroscopically smooth behavior in the strain range corresponding to a low work-hardening rate. Indeed, as can be seen in Fig. 2(b), no stress serrations can be resolved on the macroscopic scale during this stage of deformation.

Magnification of the deformation curves reveals complex patterns of small-scale stress oscillations illustrated in the insets of Figs. 2(b) and 3(b). During the initial stage corresponding to the high work-hardening rate their amplitude and shape depend in an intricate manner on both strain rate and strain. The investigation of this nonstationary behavior goes beyond the scope of the present paper. The oscillations are stabilized when the work-hardening rate falls down, as shown in the inset of Fig. 2(b). Interestingly, they display similar shapes and amplitudes (about 0.2 MPa) in a wide $\dot{\varepsilon}_a$-range, except for the highest value of $7 \times 10^{-3}$ s$^{-1}$. In this last case the fine structure of the oscillations becomes effectively smoothed because of the fast loading. The observation of such complex patterns raises two main questions: (i) Are they random or correlated? (ii) Are they related to the DSA or rather stem from another microscopic mechanism? In order to shed light on these questions, the temporal patterns will be analyzed below and compared with the results of similar analysis of the stress serrations induced by the PLC effect in the respective time intervals characterized by a stabilized jerky flow and a low work-hardening rate.

3.4. Quantitative analysis of temporal patterns

Fig. 4 presents examples of statistical distributions of the size of stress fluctuations in CG and UFG materials. The statistical behavior of the PLC effect is well known [10–14]. The histogram plotted in

Fig. 3. Magnified portions of curves 1 of Fig. 2. The insets correspond to higher magnification revealing complex patterns of stress oscillations. The arrow in the left plot marks the critical strain for the onset of the PLC instability.

Fig. 4. Examples of distributions of amplitudes of stress fluctuations for CG (upper line) and UFG (bottom line) material. The value of $n$ gives the number of events with the size within the corresponding bin. The $\dot{\varepsilon}_a$-values are as follows: (a) $2 \times 10^{-3}$ s$^{-1}$; (b) $7 \times 10^{-3}$ s$^{-1}$; (c) $2 \times 10^{-3}$ s$^{-1}$; (d) $1 \times 10^{-3}$ s$^{-1}$. 
Since the first observation of such scale-free statistics of the PLC effect in Ref. [10], it is generally ascribed to the so-called self-organized criticality (SOC), a concept introduced to explain avalanche-like dynamics [27]. When the strain rate is decreased, an inherent scale of stress drops progressively appears, as reflected in the occurrence of a bell-shaped histogram illustrated in Fig. 4(a), which is usually the more pronounced the smaller is the strain rate.

The comparison of statistical behavior of the PLC serrations and the accompanying acoustic emission [26], as well as analysis of continuous acoustic emission waveforms [28], led to a hypothesis that this transition is due to a concurrency between SOC and the phenomenon of synchronization of the dynamics of nonlinear systems composed of many elements [29]. The latter phenomenon leads to behavior of relaxation-oscillation type and, consequently, imposes an inherent time and size scale. It should be emphasized that these phenomena represent two sides of the same problem of self-organization in complex nonlinear systems. However, the underlying mechanisms can give rise to different dynamical regimes, depending on the nature of the nonlinearity and spatial correlation between the system components.

It should be mentioned that in addition to the bell-shaped histogram, the plot in Fig. 4(a) displays a high probability of very small stress fluctuations corresponding to the bin near the origin. In [26], the separate analysis of the deep PLC serrations and low-amplitude oscillations showed that the latter cannot be attributed to some kind of random noise because they are also described by a power law. Similar analysis performed in the present work confirmed this observation, which proves a nonstochastic nature of low-amplitude stress fluctuations described above.

Figs. 4(c) and (d), and 5(b) present results of a similar analysis of low-amplitude oscillations observed in UFG samples, except for the case of the over-smoothened deformation curves obtained at the highest strain rate of \(7 \times 10^{-3}\) s\(^{-1}\). The general trend of deviation from the power law when the strain rate is decreased remains valid in this case, too. However, it occurs that the tendency to scale-free behavior is much stronger than for the PLC effect. Indeed, the histograms remain more or less monotonically descending even at the smallest strain rate of \(2 \times 10^{-3}\) s\(^{-1}\), as can be seen in Fig. 4(c). It displays reasonably good power-law dependencies for higher strain rates, as illustrated in Figs. 4(d) and 5(b).

The major conclusion following from these results is that the low-magnitude stress fluctuations are not of random nature. In the spirit of the above hypothesis, the proneness to power-law behavior may be interpreted as being due to less efficient synchronization of deformation processes in UFG samples. Additional arguments in favor of this hypothesis are provided by the multifractal analysis presented below.

Examples of partition functions \(S_q(\delta t)\) presented in Fig. 6 for an UFG sample confirm the conclusion on the nonrandom character of stress fluctuations (cf. [14,15,30] for CG materials). Indeed, a fan of linear dependencies corresponding to the generalized fractal dimensions \(D(q)\). The slope of such dependencies renders the values of the generalized fractal dimensions \(D(q)\). Vertical dashed lines show the scaling interval used to calculate \(D(q)\) dependencies.

The comparison of these results with similar dependencies for UFG specimens allow for two assumptions. First, the curves

\[D(\delta t) = \frac{\log_{10}(S_q)}{\log_{10}(\delta t)}\]

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obtained at different strain rates lay in the vicinity of each other (curves 2 and 4). Not only this result agrees with the observation that the corresponding stress fluctuations are similar in size and shape, but it moves such comparison from a qualitative to a quantitative level. Second, the $D(q)$ dependencies occupy an intermediate position between those found in the CG case. This result confirms the above conjecture of less efficient synchronization of deformation processes in UFG samples but nevertheless, corresponds to relatively homogeneous behavior, as compared with that found for type A serrations (curve 3).

3.5. Remarks on the deformation mechanisms

The proposed interpretation of the data of statistical and multifractal analysis can be justified in the framework of generic predictions of the models of self-organization in complex dynamical systems. According to [29], the SOC is favored by a high strength of spatial coupling between local strain heterogeneities and week nonlinearity of the driving force, while the phenomenon of synchronization occurs in the opposite case. As the lesser capacity of plastic flow impedes the effective relaxation of internal stresses, which are caused by local strain incompatibilities generated during deformation, the strength of the spatial coupling is likely to become higher after grain refinement. Indeed, the UFG material exhibits little ductility, chiefly because of its very low strain hardening capability. Further, according to the data in Fig. 2, the strain-rate sensitivity of stress, reflecting the nonlinearity of the driving force, is close to zero in the UFG material, in contrast to the negative values characterizing the CG material. It follows that the synchronization of the dislocations dynamics will be hindered in the UFG state. It is also noteworthy that as far as the dislocations synchronization is a necessary condition for the occurrence of macroscopic stress serrations, its obstruction may be one of the factors leading to the observed suppression of the PLC effect.

As far as the microscopic mechanism of low-amplitude stress fluctuations in the UFG material is concerned, the answer is not clear yet. On the one hand, the similarity of the nonstationary temporal patterns during the initial portions of deformation curves of both kinds of specimens (see Fig. 3) testifies that the DSA mechanism may still be important in UFG materials. Nevertheless, the proposed explanation of the further statistical and multifractal behavior is virtually independent of the assumption of a microscopic mechanism. Consequently, it does not allow suggesting whether the small fluctuations observed in the tests on the UFG samples are related to the DSA mechanism or caused by pure dynamical mechanisms of collective motion of dislocations. With this concern in view, attention can be drawn to recent investigations of the small-scale plasticity which showed that even in the case of pure materials the plastic flow is inherently heterogeneous and intermittent on a fine scale revealed either by high-resolution techniques, such as the acoustic emission method [32], or by extreme reduction of the specimen dimensions [33]. The totality of the literature data testifies that such intermittency is always characterized by power-law statistics.

4. Conclusions

In summary, the results presented permit the following conclusions to be drawn:

- The extreme grain refinement by ECAP processing of alloys may result in effective suppression of the PLC effect.
- Stress fluctuations with amplitude about two orders of magnitude smaller than the PLC serrations are still discernible on the deformation curves. While being dwarfed under conditions when large scale PLC serrations prevail, they come to the fore when these are suppressed. The statistical and multifractal analyses of such small amplitude fluctuations confirmed their nonrandom character.
- The comparison of these results with similar data for the PLC effect can be explained qualitatively in terms of the concurrency between the phenomena of self-organized criticality and synchronization in complex systems. More specifically, it is conjectured that the extreme grain refinement hinders the synchronization of the dislocation dynamics. Moreover, the obstruction of the synchronization of the events of discontinuous dislocation motion may be an important factor explaining the disappearance of the PLC instability.
- In order to obtain better insight in both the mechanism of suppression of the PLC effect and the mechanism of the low-amplitude stress fluctuations in the UFG alloy, various tracks can be envisaged. In particular, in future work it would be of interest to relate the observed temporal patterns to spatial patterns associated with the local strain localizations.

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