

# The influence of shearable and nonshearable precipitates on the Portevin-Le Chatelier behavior in precipitation hardening AlMgScZr alloys

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## The influence of shearable and nonshearable precipitates on the

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## Portevin-Le Chatelier behavior in precipitation hardening

3	AlMgScZr alloys
4	Han Chen <sup>a</sup> , Zhe Chen <sup>a*</sup> , Gang Ji <sup>b</sup> , Shengyi Zhong <sup>c*</sup> , Haowei Wang <sup>a</sup> , András Borbély <sup>d</sup> , Yubin Ke <sup>e</sup> ,
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17	Abstract: The well-known mechanisms of interaction between precipitates and
18	dislocations are shearing (for shearable precipitates) and bypassing mechanisms (for
19	nonshearable precipitates). The transition from shearable to nonshearable precipitates
20	in precipitation hardening alloys leads to changes of dislocation motion mode and
21	dislocation multiplication behavior, which inevitably causes different PLC behaviors.
22	In this study, we systematically investigate the influence of shearable and
23	nonshearable Al <sub>3</sub> (Sc, Zr) precipitates on PLC behaviors by experimental
24	characterization for precipitation hardening AlMgScZr alloys. We analyze the onset
25	strain, critical strain-rate range, serration amplitude, and propagation behavior of PLC
26	bands in detail for AlMgScZr alloys with shearable and nonshearable precipitates,
27	respectively. We find that the transition from shearable to nonshearable precipitates
28	changes the PLC band propagation behavior, decreases the magnitude of serration
29	amplitude, expands the strain-rate range as well as decreases the critical strain rate

and inverse behavior (the critical strain decreases with the increase of strain rate) regimes due to the different dislocation-precipitate interactions. Besides, the transition from shearable to nonshearable precipitates increases the onset strain at normal behavior while decreases the onset strain at inverse behavior depending on the different roles of precipitates in the solute-dislocation interaction. Finally, we reveal the nature of influence of different dislocation-precipitate interactions on PLC behavior considering different strengthening mechanisms based on quantitative characterization on precipitates and dislocation density.

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## Keywords: Aluminium alloy, Precipitates, Portevin-Le Chatelier effect, Strengthening mechanism, Dislocations

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### 1. Introduction

Portevin-Le Chatelier (PLC) is a kind of dynamic strain ageing (DSA) 44 phenomenon due to the interaction between diffusing solute atoms and mobile 45 dislocations, which has been found in many alloy systems, particularly in AlMg alloys 46 [McCormick, 1972a; Van den Beukel, 1975; Estrin and Kubin, 1990; Kubin and 47 Estrin, 1990; Robinson, 1994; Hähner, 1997; Lebyodkin et al., 2000; Picu, 2004; 48 Fressengeas et al., 2005; Antolovich and Armstrong, 2014; Aboulfadl et al., 2015; 49 Swaminathan et al., 2015; Zhang et al., 2017; Tsai et al., 2019]. On the atomic level, 50 51 the PLC phenomenon is caused by the diffusion of solute atoms [Cottrell and Bilby, 1949; Yoshinaga and Morozumi, 1971a, 1971b; Aifantis, 1987; Estrin and Kubin, 52 53 1990; Picu, 2004; Curtin et al., 2006; Soare and Curtin, 2008a, 2008b; Aboulfadl et al., 2015]. The dislocations are efficiently pinned by solutes in direct relation with the 54 55 solute which has segregated to its core. The longer the waiting time (i.e. the lower the imposed strain rate) of a dislocation between two jumps, the higher the solute 56 concentration at dislocation core and therefore the higher the resulting flow stress 57 [McCormick, 1972a; Estrin and Kubin, 1990, 1991, 1995; Kubin and Estrin, 1985, 58 59 1990; Curtin et al., 2006]. The interaction between dislocations and solute atoms can 60 result in a decrease of the flow stress associated to an increase of the imposed strain rate and thus to a negative strain rate sensitivity (SRS) [Van den Beukel, 1975; 61 Mulford and Kocks, 1979; Louat, 1981; Kubin et al., 1988; Estrin and Kubin, 1990, 62

1995; Kubin and Estrin, 1990; Picu, 2004; Curtin et al., 2006]. On a macroscopic scale, the PLC phenomenon is generally manifested by sudden localizations of the plastic strain in transient deformation bands, leading to jerky flow features on the stress strain curves [Zhang et al., 2001; Kok et al., 2003; Zhang et al., 2005; Benallal et al., 2008; Zavattieri et al., 2009; Manach et al., 2014; Klusemann et al., 2015; Shibkov et al., 2016; Yuzbekova et al., 2017; Yang et al., 2018; Zhemchuzhnikova et al., 2018; Ren et al., 2021]. When PLC occurs, each serration of the deformation curve corresponds to the development of localized deformation bands [Klusemann et al., 2015; Yuzbekova et al., 2017; Zhemchuzhnikova et al., 2018]. The nucleation and propagation of deformation bands have been proved to have a close relation to jerky flow for many materials with PLC behavior, especially in AlMg alloys [Zhang et al., 2001; Ait-Amokhtar et al., 2006a, 2006b; Zavattieri et al., 2009; Manach et al., 2014; Klusemann et al., 2015; Yang et al., 2018]. Three major types of PLC bands, namely continuous propagating type A, hopping type B and non-propagating type C bands are defined based on the serrated curves [Pink and Grinberg, 1981; Robinson and Shaw, 1994; Jiang et al., 2007; Yilmaz, 2011; Hu et al., 2012; Zhang et al., 2017]. The PLC bands generated during deformation leave undesirable traces on the surface of sheet products during deep drawing restricting the commercial application of some alloys [Dierke et al., 2007; Böhlke et al., 2009; Yilmaz, 2011; Duan et al., 2021]. Moreover, the ductility will be decreased due to the appearance of PLC [Kang et al., 2006; Aretz, 2007; Halim et al., 2007; Zhang et al., 2012; Alinaghian et al., 2014; Keralavarma et al., 2014; Rousselier and Quilici, 2015]. Undoubtedly, the PLC effect is expected to be diminished by designing specific alloy compositions or by controlling processing conditions. Nanostructured precipitates are beneficial to eliminate the PLC effect, which already has been reported previously [Riley and McCormick, 1977; Hayes and Hayes, 1982, 1984; Wert and Wycliffe, 1985; Pink, 1989; Kumar and McShane, 1993; Kumar et al., 1996; Thevenet et al., 1999; Nalawade et al., 2008; Wang et al., 2016; Cai et al., 2017]. Pink and Thevenet et al. [Pink, 1989; Thevenet et al., 1999] proved that the jerky flow in AlZnMg alloy would be restrained by precipitates, especially the appearance of GP-zones could increase the onset strain of PLC significantly. Kumar and McShane [Kumar and McShane, 1993] stated that the transformation of metastable precipitates (from coherent to incoherent state) in the process of ageing can lead to the disappearance of PLC in AlLi alloys. However, some scholars underlined that solute concentration in the

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matrix decreased due to precipitation, which suppressed PLC [Riley and McCormick, 1977; Wert and Wycliffe, 1985; Nalawade et al., 2008]. There are others who emphasized that dislocations shearing of coherent precipitates directly resulted in serrations on the flow stress curve of AlLi alloys [Kumar and Pink, 1994, 1997; Kumar, 1995; Pink et al., 2000]. While some people demonstrated that dislocations shearing precipitates would not lead to jerky flow in the AlLi or in the Ni-based superalloy [Chmelik et al., 1998; Wang et al., 2016]. In the mentioned studies [Riley and McCormick, 1977; Hayes and Hayes, 1982, 1984; Wert and Wycliffe, 1985; Pink, 1989; Kumar and McShane, 1993; Kumar and Pink, 1994; Kumar et al., 1995; Kumar et al., 1996; Chmelík et al., 1998; Thevenet et al., 1999; Nalawade et al., 2008; Wang et al., 2016; Cai et al., 2017], the solute concentration in the matrix decreases when the precipitates appear, so it is difficult to distinguish whether the change of PLC behavior is caused by precipitates or solute concentration variations. Although the aforementioned works have focused on the effect of nanosized precipitates on the PLC behavior, they do not focus on the influence of precipitates on the propagation behavior of PLC bands. Only recently, Cai et al. and Wang et al. indicated that type A bands can turn into type B, and finally became type C with increasing precipitate volume fraction in Ni-based alloys [Cai et al., 2015, 2017; Wang et al., 2019]. Cai et al. explained that these changes were due to a change in the plastic deformation mechanism, which turned from dislocation slip to stacking fault generation [Cai et al., 2017]. Obviously, this mechanism cannot be applied in the AlMg system of the present study. On the other hand, to the authors' knowledge, no attention is paid on the influence of precipitate size (i.e. the effect of different precipitate-dislocation interactions) on PLC behavior without considering the variation of solute concentration. This impedes drawing clear conclusions about the influence of precipitates on the PLC behavior. The well-known mechanisms between precipitates and dislocations are shearing (for shearable precipitates) and bypassing mechanisms (for nonshearable precipitates) [Ardell, 1985; Gladman, 1999; Knipling et al., 2010, 2011; Hull and Bacon, 2011; Fan et al., 2018; Ming et al., 2018; Barnett et al., 2019; Xu et al., 2019; Chen et al., 2021]. The influence of precipitates on the PLC behavior lies in revealing the

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mechanisms of appearance of precipitates on existing dislocation-dislocation and

dislocation-solute interactions. In view of current problems, we design the AlMgScZr

alloy system with the Al<sub>3</sub>(Sc, Zr) precipitates on AlMg matrix to quantitatively study the effects of shearable and nonshearable precipitates on PLC behavior with the aid of experimental characterization in this study. The advantage of this alloy is that the solute Mg concentration keeps unchanged when Al<sub>3</sub>(Sc, Zr) precipitates occur, which avoids the influence of solute concentration variation on PLC (It is because the interaction of Mg atoms and dislocations is the one which leads to PLC phenomenon). The shearable and nonshearable Al<sub>3</sub>(Sc, Zr) precipitates can be obtained through controlling heat treatment, and their quantitative characterization can be easily obtained by transmission electron microscopy (TEM) and small angle neutron scattering (SANS) techniques due to its good uniform dispersion, spherical shape and thermal stability [Fuller et al., 2003; Clouet et al., 2006; Voorhees, 2006; Deschamps et al., 2007; Deschamps and Geuser, 2011; Chen et al., 2021]. In this study, we focus our attention on effects of different dislocation-precipitate interactions (shearing and bypassing mechanisms) on PLC behavior including propagation behavior of PLC band without considering the variation of solute concentration, which has not been reported to our knowledge. We will demonstrate the reason of the influence of dislocation-precipitate interaction on PLC behavior with considering different strengthening mechanisms based on the quantitative characterization on dislocation density measured by synchrotron radiation X-ray diffraction.

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#### 2. Materials and methods

Two cast AlMg and AlMgScZr alloys were used. The chemical compositions in wt.% of AlMg and AlMgScZr are Al-5.7%Mg and Al-5.4%Mg-0.2%Sc-0.15%Zr, respectively. The AlMg alloy was first homogenized at 813 K for 24 h and then extruded at 623 K with a ratio of 10:1. It is known that Al<sub>3</sub>(Sc, Zr) precipitates nucleate at aging temperatures higher than 598 K [Knipling et al., 2010, 2011], for which reason the cast AlMgScZr alloys were first aged at 623 K, 773 K for 3h and at 813 K for 24h correspondingly, with the purpose of obtaining shearable and nonshearable Al<sub>3</sub>(Sc, Zr) precipitates. After aging, the AlMgScZr alloys were also extruded at 623 K with an extrusion ratio of 10:1.

TEM samples were extracted from the AlMg and AlMgScZr extrusion specimens, which were polished mechanically, followed by electrochemical polishing by using a twin-jet polishing unit with a mixed solution of 1/3 methanol and 2/3 nitric acid. TEM

experiments were carried out using a high-resolution Cs-corrected FEI Themis microscope operating at 300 kV and a JOEM-2100F operating at 200 kV. The quantitative characterization on precipitates is necessary in the discussion of different dislocation-precipitate interactions on PLC behaviors, which was obtained by SANS. The SANS experiment was performed on the SANS instrument at China Spallation Neutron Source (CSNS) [Ke et al., 2018]. The detailed information about SANS and data fitting process can be found in reference [Chen et al., 2021].

The tensile deformation of heat-treated AlMg and AlMgScZr alloys was carried out at room temperature (298 K) using strain rates between  $5\times10^{-1}$ – $1.0\times10^{-6}$  s<sup>-1</sup>. The gauge section of tensile specimens with dog-bone shape was 15×3×2 mm<sup>3</sup>. The dislocation densities of extruded AlMg and AlMgScZr alloys were determined by high resolution X-ray diffraction line profile analysis (XLPA), which were measured by synchrotron radiation X-ray diffraction (SRXRD) conducted at BL14B1 beamline in Shanghai Synchrotron Radiation Facility (SSRF). The average dislocation density was determined from the asymptotic behavior of the second and fourth-order restricted moments [Borb é ly and Groma, 2001] of the intensity distribution corresponding to the 200, 220 and 311 diffraction peaks of the samples. the dislocation density in this study is determined by variance method. The asymptotic behavior of the variance in our method does not depend on dislocation arrangement [Kalácska et al., 2017]. This is because the asymptotic behavior is only influenced by the strain field in the vicinity of dislocations and not by the dislocation arrangement (dislocations far away). This is a well-known property of the Fourier transform linking small distances in the real space (close to dislocations) to large distances in the wave vector space (the tails of the diffraction peak). Therefore, the determined total dislocation density by SRXRD is reliable, although the distribution of dislocation is non-uniform in AlMgScZr-623K/3h alloy (seen discussion section). The detailed information about variance method is shown in supplementary materials.

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## 3. Results and analysis

#### 3.1 Quantitative characterization of Al<sub>3</sub>(Sc, Zr) precipitates

Fig. 1 shows dark-field TEM images of Al<sub>3</sub>(Sc, Zr) precipitates and the corresponding selected area electron diffraction (SAED) patterns for

AlMgScZr-623K/3h, AlMgScZr-773K/3h and AlMgScZr-813K/24h alloys. The precipitates have a spherical shape and are distributed relatively uniformly in the aluminum matrix (Fig. 1). Fig. 2 shows the curves of scattering intensity I versus scattering vector q captured by SANS and the corresponding raw two-dimensional scattering data for these three kinds of AlMgScZr alloys. The mean radius, volume fraction and precipitate inter-distance obtained from SANS are shown in Table 1. The results in Table 1 reveal that precipitate size increases with increasing aging temperature and mean radius is about 2.5, 10.2 and 22.6 nm for AlMgScZr-623K/3h, AlMgScZr-773K/3h, AlMgScZr-813K/24h alloys. The corresponding volume fraction of these three samples is about 0.46%, 0.42% and 0.40%, which remains practically unchanged considering the error of the evaluation of about 10%. The results also show that the increase of precipitate size leads to an increase of inter-precipitate distance (Table 1) [Knipling et al., 2010, 2011]. The quantitative information on Al<sub>3</sub>(Sc, Zr) (average radius and volume fraction) obtained by TEM and SANS is close to the previous results for the Al-Sc-Zr systems [Seidman et al., 2002; Fuller et al., 2003; Clouet et al., 2005; Marquis and Seidman, 2005; Deschamps et al., 2007; Knipling et al., 2010; Taendl et al., 2016]. It can be seen from Table 1 that the precipitate radius for AlMgScZr-623K/3h alloy is smaller than 3 nm (the critical radius between shearable and nonshearable precipitates is about 3-4 nm [Fazeli et al., 2008; Knipling et al., 2010; Lai et al., 2013; Okle et al., 2019; Chen et al., 2021], thus the precipitates in this alloy are defined as shearable precipitates. Analogously, the precipitates in AlMgScZr-773K/3h and AlMgScZr-813K/24h alloys are defined as nonshearable precipitates. In order to verify that the above definition on precipitates in the three AlMgScZr alloys are reasonable, their strengthening effect are quantified by comparing model prediction and experimental results, as discussed in 4.1 section. The model prediction and experimental results are in good agreement within experimental accuracy, further demonstrating the precipitates in AlMgScZr-623K/3h alloy are shearable as well as in AlMgScZr-773K/3h and AlMgScZr-813K/3h alloys are nonshearable.

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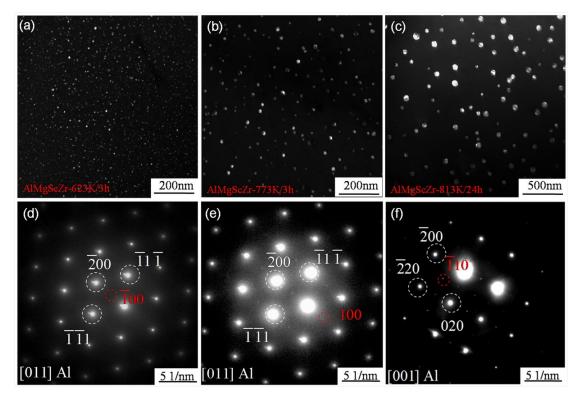


Fig. 1 Characterization on Al<sub>3</sub>(Sc, Zr) precipitates by dark-field TEM. The dark-field TEM images of Al<sub>3</sub>(Sc, Zr) precipitates of (a) AlMgScZr-623K/3h alloy, (b) AlMgScZr-773K/3h alloy and (c) AlMgScZr-813K/24h alloy; (d-f) the corresponding SAED patterns of (a-c). The extracted spots for above dark-field images (a-c) are  $(\overline{1}00)$ , (100) and  $(\overline{1}10)$ , respectively.

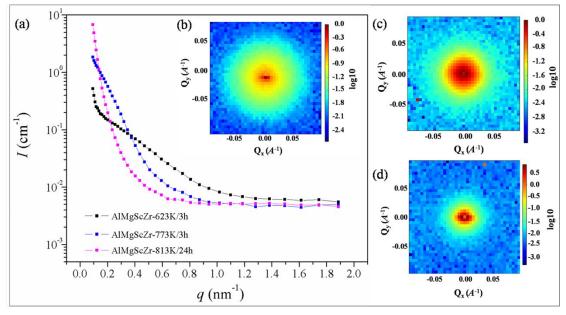


Fig. 2 Quantitative characterization on precipitates by SANS. (a) Scattering intensity I versus scattering vector q for AlMgScZr-623K/3h [Chen et al., 2021], AlMgScZr-773K/3h [Chen et al., 2021] and AlMgScZr-813K/24h alloys; (b-d) the corresponding raw two-dimensional scattering data for AlMgScZr-623K/3h,

Table 1 Quantitative information on precipitates provided by SANS and precipitate attribute. The critical precipitate size between shearable and nonshearable precipitates is about 3-4 nm. When the radius of precipitates is smaller than 3 nm, the precipitates are defined shearable precipitates. When the radius of precipitates is higher than 4 nm, the precipitates are defined as nonshearable precipitates.

Camples	Average	Volume	Inter-distance	Precipitate	
Samples	radius (nm)	fraction (%)	(nm)	attribute	
AlMg	-	-	-	without precipitates	
AlMgScZr-623K/3h	$2.5 \pm 0.7$	$0.46 \pm 0.05$	49.4	shearable precipitates	
AlMgScZr-773K/3h	$10.2 \pm 2.2$	$0.42 \pm 0.04$	211.7	nonshearable precipitates	
AlMgScZr-813K/24h	$22.6 \pm 4.9$	$0.40 \pm 0.03$	481.5	nonshearable precipitates	

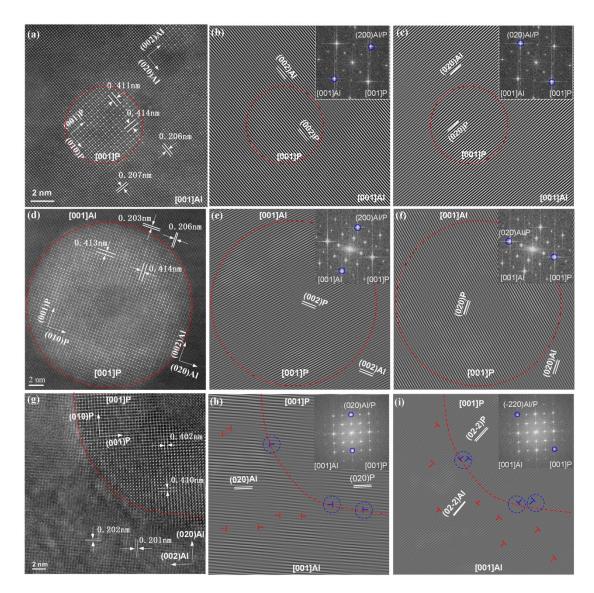


Fig. 3 HRTEM and IFFT images of shearable and nonshearable Al<sub>3</sub>(Sc, Zr) precipitates: (a-c) shearable precipitates (in the AlMgScZr-623K/3h sample); (d-f) nonshearable and coherent precipitates (in the AlMgScZr-773K/3h sample); (g-i) nonshearable and semi-coherent precipitates (in the AlMgScZr-813K/24h sample). (a), (d) and (g) are HRTEM images, (b-c), (e-f) and (h-i) are corresponding IFFT images. The red dashed lines represent the boundary between aluminum matrix and precipitates. The red "T" denotes dislocations in the aluminum matrix, while the blue "T" refers to interfacial dislocations. No interfacial dislocations are observed for AlMgScZr-623K/3h and AlMgScZr-773K/3h samples (coherent precipitates) and lots of interfacial dislocations are observed for AlMgScZr-813K/24h sample (semi-coherent precipitates). The (002)Al, (020)Al, (001)P, (010)P refers to the (002), (020) crystallographic planes of aluminum matrix and (001), (010) crystallographic planes of the precipitates, respectively; [001]P and [001]Al represent the crystal zone axis for the precipitate and matrix, respectively.

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Figs. 3(a), (d) and (g) show high resolution TEM (HRTEM) images of shearable (AlMgScZr-623K/3h alloy) nonshearable and Al<sub>3</sub>(Sc, Zr) precipitates (AlMgScZr-773K/3h and AlMgScZr-813K/24 alloys). Their Inverse Fast Fourier Transform (IFFT) reveals that the Al/Al<sub>3</sub>(Sc, Zr) interface is free of dislocations for the shearable precipitates (Figs. 3(b-c)) and nonshearable precipitates of AlMgScZr-773K/3h alloy (Figs. 3(e-f)), manifesting that the precipitates are coherent with aluminum matrix. However, lots of interfacial dislocations are observed for the nonshearable precipitates of AlMgScZr-813K/24 alloy (Figs. 3(h-i)) demonstrating that the precipitates are semi-coherent with aluminum matrix. According to previous literature [Iwamura and Miura, 2004], the driving force required for the formation of interfacial dislocations mainly results from the elastic energy caused by the lattice misfit. Hence, the appearance of interfacial dislocations indicates loss of coherency [Marquis and Seidman, 2001]. It has been shown for the case of Al-Sc alloys that precipitates are coherent with the aluminum matrix when their average radius rave<15 nm, and become semi-coherent when r<sub>ave</sub>>40 nm [Iwamura and Miura, 2004]. In the intermediate stage (15<raye<40 nm), there are coexisting coherent and semi-coherent precipitates [Iwamura and Miura, 2004]. Therefore, in this study, precipitates are found to be coherent completely with matrix for AlMgScZr-623K/3h and AlMgScZr-773K/3h alloys, but the coherent and semi-coherent precipitates coexist

<sup>&</sup>lt;sup>1</sup> Note: For AlMgScZr alloy, the additions of Mg and Zr elements may increase the critical radius between coherent and semi-coherent precipitate due to Mg and Zr elements reducing the lattice mismatch between the matrix and precipitate.

#### 3.2 Deformation curves of AlMg and AlMgScZr alloys

Fig. 4 shows the engineering stress strain curves of the AlMg and three kinds of AlMgScZr alloys deformed at different strain rates. For the AlMg alloy, the PLC phenomenon occurs in a large interval of strain rate spanning from 1.0×10<sup>-1</sup> s<sup>-1</sup> to 1.0×10<sup>-6</sup> s<sup>-1</sup> (Fig. 4(a)). The deformation curves of AlMgScZr-623K/3h, AlMgScZr-773K/3h and AlMgScZr-813K/24h alloys are displayed in Figs. 4(b), (c) and (d). The corresponding strain-rate ranges for the occurrence of PLC phenomenon are  $1.0 \times 10^{-1} - 2.5 \times 10^{-4}$  s<sup>-1</sup>,  $1.0 \times 10^{-1} - 1.0 \times 10^{-5}$  s<sup>-1</sup> and  $1.0 \times 10^{-1} - 1.0 \times 10^{-6}$  s<sup>-1</sup>. Fig. 5 shows the magnified figures of serrated flow of AlMg and three AlMgScZr alloys. From Fig. 5, the PLC phenomenon is manifested by approximately upward stress steps, followed by a stress plateau when strain rate is higher than 1.0×10<sup>-2</sup> s<sup>-1</sup>, which is similar to the previous studies [Böhlke et al., 2009; Duan et al., 2021]. When strain rate is less than  $1.0 \times 10^{-2}$  s<sup>-1</sup>, the PLC appears like saw-like serrations. Type A, B and C PLC bands are identified from the stress strain curves, and they appear at high, middle and low strain rates respectively (the mixed type A+B is also observed) both for AlMg and three AlMgScZr alloys. Upward steps, locking serrations and unlocking serrations are visible in magnified regions of the deformation curves (Fig. 5).

Figs. 6(a-d) show the variation of critical strain versus strain rate for AlMg and three AlMgScZr alloys. It can be seen that the relation between the critical strain for the onset of PLC and strain rate is non-monotonic for AlMg and three AlMgScZr alloys. Both the "normal" behavior (the critical strain for the occurrence of PLC increases with the increase in strain rate) and "inverse" behavior (the critical strain for the onset of PLC decreases with the increase in strain rate) are observed for these four materials. Moreover, the type A and A+B/B bands belongs to normal behavior, while the type C band belongs to inverse behavior (Figs. 6(a-d)). The critical strain rate for normal and inverse behaviors, the propagation behaviors of PLC bands as well as the serration amplitude all change with the shearable precipitates becoming nonshearable precipitates, which will be analyzed in details in the following section.

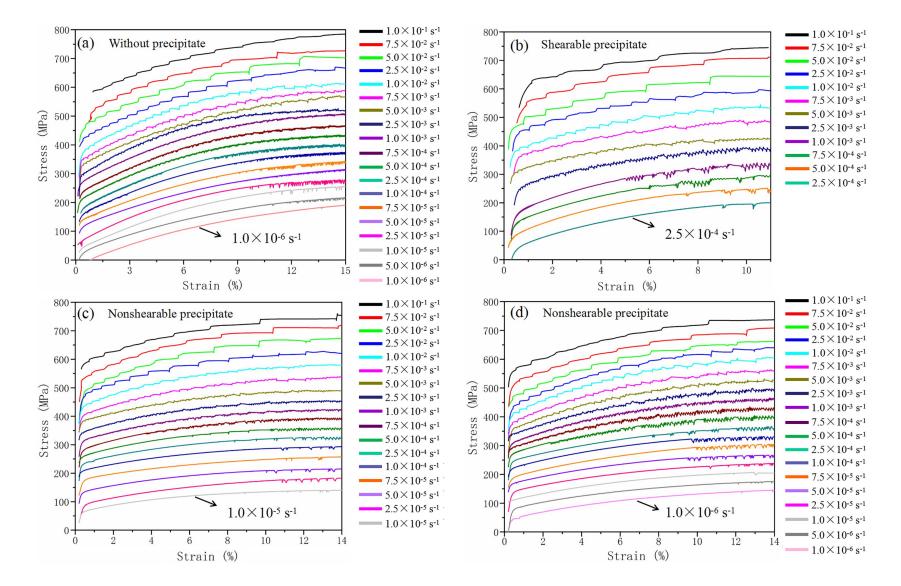
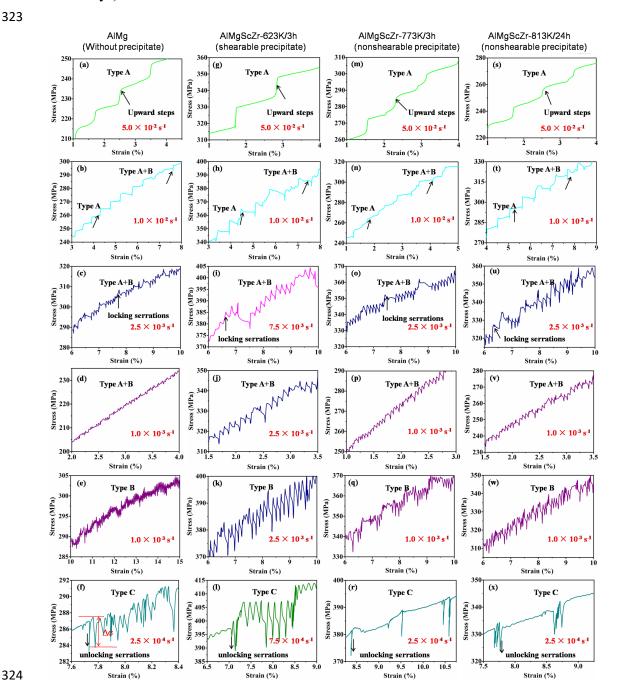


Fig. 4 Engineering stress-strain curves of: (a) AlMg (without precipitates), (b) AlMgScZr-623K/3h (shearable precipitates), (c) AlMgScZr-773K/3h (nonshearable precipitates), (d) AlMgScZr-813K/24h (nonshearable precipitates). For a better view, all curves are vertically displaced by 30-50 MPa and the end of the curves is cut. The upper critical strain rate for the occurrence of PLC is 1.0×10<sup>-1</sup> s<sup>-1</sup> for AlMg and three AlMgScZr alloys. The lower critical strain rates for the occurrence of PLC are  $2.5 \times 10^{-4} \text{ s}^{-1}$ ,  $1.0 \times 10^{-5} \text{ s}^{-1}$  for AlMgScZr-623K/3h, AlMgScZr-773K/3h alloys. The lower critical strain rate for the occurrence of PLC is smaller than 1.0×10<sup>-6</sup> s<sup>-1</sup> for AlMg and AlMgScZr-813K/24 alloys (Due to the limited tensile condition, we cannot determine the detailed lower values of strain rate for the occurrence of PLC for these two alloys).



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- Fig. 5 Magnified figure of jerky flow for the AlMg and three AlMgScZr alloys. (a-f)
  AlMg alloy; (g-l) AlMgScZr-623K/3h alloy; (m-r) AlMgScZr-773K/3h alloy; (s-x)
  AlMgScZr-813K/24h alloy. For every column, type A (upward steps) first turns to
  type A+B, then to type B and final to type C (unlocking serrations) with the decrease
  of strain rate (from top to bottom). The "Δσ" in the (f) denotes the serration amplitude,
- which is described in 3.3.3 section.

#### 3.3 PLC behavior of AlMg and AlMgScZr alloys

## 3.3.1 Influence of the precipitates on onset strain and strain-rate range of PLC

The variations of critical strain and critical strain-rate range of PLC in AlMg and three AlMgScZr alloys is illustrated in Fig. 6 and Table 2. For the AlMg alloy, the strain rate dependence of critical strain exhibits normal behavior in the  $5.0 \times 10^{-4} - 1.0 \times 10^{-1}$  s<sup>-1</sup> range and an inverse behavior in the  $1.0 \times 10^{-6} - 5.0 \times 10^{-4}$  s<sup>-1</sup> range, as shown in Fig. 6(a) and Table 2. This non-monotonous behavior is also observed for AlMgScZr alloy with precipitates, but the onset strain and strain-rate ranges of PLC change due to the presence of precipitates (Figs. 6(b-d), (e-l) and Table 2). In the normal behavior, shearable precipitates decrease the onset strain of PLC obviously, but this onset strain increases again with the shearable precipitates being replaced by nonshearable precipitates (Figs. 6(e-h)). However, the precipitates have an opposite effect on the onset strain in the inverse behavior regime. That is the shearable precipitates first increase the onset strain significantly, then the onset strain decreases when shearable precipitates become nonshearable precipitates (Figs. 6(i-l)).

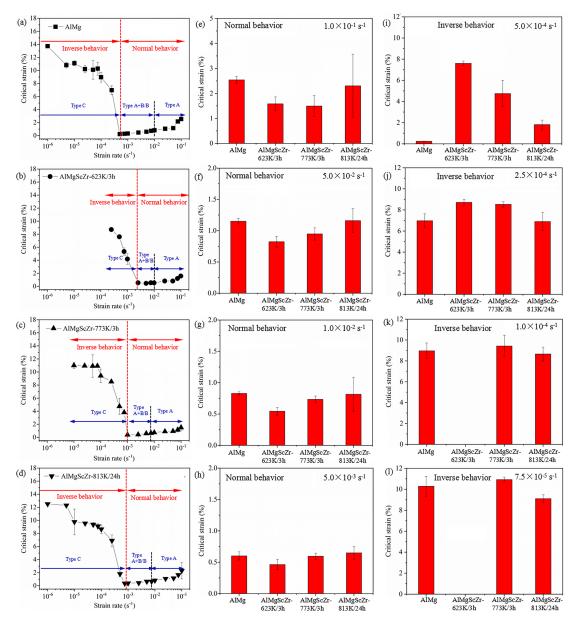


Fig. 6 (a-d) The variation of critical strain versus strain rate for AlMg (without precipitates), AlMgScZr-623K/3h (shearable precipitates), AlMgScZr-773K/3h (nonshearable precipitates) and AlMgScZr-813K/24h (nonshearable precipitates). The black dashed lines are the boundary lines between type A and type A+B/B bands; the red dashed lines also are the boundary lines between normal and inverse behaviors; (e-h) comparison of critical strain for above alloys at different strain rates for normal behavior: (e)  $1.0 \times 10^{-1}$  s<sup>-1</sup>, (f)  $5.0 \times 10^{-2}$  s<sup>-1</sup>, (g)  $1.0 \times 10^{-2}$  s<sup>-1</sup>, (h)  $5.0 \times 10^{-3}$  s<sup>-1</sup>; (i-l) comparison of critical strain for above alloys at different strain rates for inverse behavior: (i)  $5.0 \times 10^{-4}$  s<sup>-1</sup>, (j)  $2.5 \times 10^{-4}$  s<sup>-1</sup>, (k)  $1.0 \times 10^{-4}$  s<sup>-1</sup> (PLC disappears for AlMgScZr-623K/3h alloy), (l)  $7.5 \times 10^{-5}$  s<sup>-1</sup> (PLC disappears for AlMgScZr-623K/3h alloy).

Samples	Normal behavior range (s <sup>-1</sup> )	Inverse behavior range (s <sup>-1</sup> )	Critical strain rate (s <sup>-1</sup> )
AlMg (without precipitates)	$5.0 \times 10^{-4} - 1.0 \times 10^{-1}$	1.0×10 <sup>-6</sup> –5.0×10 <sup>-4</sup>	5.0×10 <sup>-4</sup>
AlMgScZr-623K/3h (shearable precipitates)	$2.5 \times 10^{-3} - 1.0 \times 10^{-1}$	2.5×10 <sup>-4</sup> -2.5×10 <sup>-3</sup>	2.5×10 <sup>-3</sup>
AlMgScZr-773K/3h (nonshearable precipitates)	$1.0 \times 10^{-3} - 1.0 \times 10^{-1}$	1.0×10 <sup>-5</sup> –1.0×10 <sup>-3</sup>	1.0×10 <sup>-3</sup>
AlMgScZr-813K/24h (nonshearable precipitates)	$7.5 \times 10^{-4} - 1.0 \times 10^{-1}$	1.0×10 <sup>-6</sup> –7.5×10 <sup>-4</sup>	7.5×10 <sup>-4</sup>

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The critical strain rate for which one observes a transition between normal and inverse behaviors increases evidently when shearable precipitates are introduced. With increasing the precipitate size (from shearable to nonshearable), this critical strain rate decreases continuously and comes back to almost the value observed in the AlMg alloy when the size of nonshearable precipitate is very large (Table 2). On the other hand, by comparing AlMg and AlMgScZr-623K/3h alloys, the strain-rate range reduces from  $1.0 \times 10^{-1} - 1.0 \times 10^{-6}$  s<sup>-1</sup> to  $1.0 \times 10^{-1} - 2.5 \times 10^{-4}$  s<sup>-1</sup> with the introduction of shearable precipitates (Table 2). However, the strain-rate range expands when shearable precipitates are replaced by nonshearable precipitates. Finally this strain-rate range becomes similar to that of the AlMg alloy when the nonshearable precipitate size is very large (for AlMgScZr-813K/24h alloy). It can be seen that the influence of precipitates on strain-rate range is mainly focused on the lower critical strain rate (the lowest strain rate for the appearance of PLC). That is the first increase and then decrease of lower critical strain rate lead to narrowing and expansion of strain-rate range with the introduction of shearable precipitates and subsequent growing into nonshearable precipitates. Therefore, it is concluded that shearable precipitates inhibit the PLC phenomenon at lower strain rate, but this inhibition effect is eliminated gradually when shearable precipitates become nonshearable by coarsening.

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#### 3.3.2 Influence of the precipitates on the propagation behavior of PLC bands

It is generally accepted that with the decrease in strain rate type A bands turn into

type B and finally form type C bands [Mogucheva et al., 2016; Zhang et al., 2017]. 386 The corresponding serrations or steps of type A are periodic, and the serrations are 387 usually locking serrations [Jiang et al., 2007; Hu et al., 2012; Zhemchuzhnikova et al., 388 2015; Zhang et al., 2017] (Figs. 5(a), (g), (m) and (s)). The locking serrations are 389 characterized by an abrupt rise, followed by a drop to the general level of the 390 deformation curve. Type B bands propagate over short distances discontinuously 391 along the gauge direction and then disappear [Jiang et al., 2007; Hu et al., 2012; 392 Zhemchuzhnikova et al., 2015]. The corresponding serrations are locking and 393 394 oscillating, as shown in Figs. 5(e), (k), (q) and (w). The serrations corresponding to type C bands are drops in the stress that occur below the general level of the stress 395 strain curve and therefore are considered to be caused by dislocation unlocking 396 [McCormick et al., 1970, 1972b; Jiang et al., 2007; Fu et al., 2012; Zhemchuzhnikova 397 et al., 2015] (Figs. 5(f), (l), (r) and (x)). It should be noted that there is more evident 398 band propagation (smooth intervals) between deep stress drops of type C for 399 AlMgScZr alloys with precipitates [Zhemchuzhnikova et al., 2017] (Figs. 5(r) and 400 (x)). In this work it is found that type A bands appear in the  $1.0 \times 10^{-2} - 1.0 \times 10^{-1}$  s<sup>-1</sup> 401 range for AlMg alloy (Figs. 5(a) and 6(a)). It should be noted that in this alloy the first 402 403 emerging PLC band is of type A at the beginning of deformation, and evolves into type A+B bands when the accumulated plastic strain is high, as shown in Fig. 5(b). 404 When strain rate is decreased, mixed type A+B bands dominate the deformation 405 curves, as show in Figs. 5(c) and (d). Type B bands can be superimposed on the type 406 407 A+B bands and mainly persist at later stages of deformation [Mogucheva et al., 2016] (Figs. 5(d) and (e)). With further decrease of strain rate, type C bands appear for the 408 AlMg sample (Fig. 5(f)). Unlocking type C bands do not occur at the beginning of 409 plastic deformation, and they usually occur when plastic strain reaches a certain 410 amount. The introduction of shearable or nonshearable precipitates does not change 411 the habitual sequence of PLC bands, that is, type A bands still transform to type 412 A+B/B band and finally to type C (Figs. 5(g-1), (m-r) and (s-x)). Precipitation changes 413 only the strain-rate ranges for the appearance of PLC bands, which is depicted in Figs. 414 6(a-d). It can be seen from Fig. 6(a-d) that the type B bands of AlMg sample (in 415 5.0×10<sup>-4</sup>-2.5×10<sup>-3</sup> s<sup>-1</sup> strain-rate range) turn to type C band when shearable 416 precipitates are introduced, and the type B bands of AlMg sample (in 417 2.5×10<sup>-3</sup>–1.0×10<sup>-2</sup> s<sup>-1</sup> strain-rate range) remain. It is demonstrated that the shearable 418 precipitates increase the lower strain rate for the appearance of type C band. With 419

shearable precipitates being replaced by nonshearable precipitates, the lower strain rate for the occurrence of type C band decreases, gradually approaching to that of the AlMg alloy when precipitate size is very large (for AlMgScZr-813K/24h alloy).

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## 3.3.3 Influence of the precipitates on serration amplitude

The serration amplitude is defined as the height of stress drop, corresponding to the vertical distance between a given local maximum and the latter local minimum on the deformation curve [Pink and Grinberg, 1981; Klose et al., 2004; Zhang et al., 2017], as shown in Fig. 5(f). Fig. 7 shows the variation of the serration amplitude versus strain at normal behavior (at higher strain rate). It is found that the serration amplitude largely increases in case of shearable precipitates and decreases with shearable precipitates becoming nonshearable precipitates (Fig. 7). As the strain range for the appearance PLC is narrow for both AlMg and three AlMgScZr alloys (especially for AlMgScZr-623K/3h, Figs. 4 and 6) in inverse behavior, we do not establish the dependence of serration amplitude versus strain like Fig. 7. We only compare the serration amplitudes of four alloys at a fixed strain, as shown in Fig. 8. It can be seen that the serration amplitude increases with the presence of shearable precipitates, while it decreases when shearable precipitates become nonshearable precipitates. The variation of serration amplitude with the presence of shearable precipitates and subsequent growing into nonshearable at inverse behavior is similar to that at normal behavior. No matter for normal behavior or inverse behavior, the magnitude of the serration

No matter for normal behavior or inverse behavior, the magnitude of the serration amplitude manifests a measure for the strength of solutes locking the dislocations [Zhang et al., 2017]. Longer dislocation waiting times are associated with lower nominal strain rates, which allow for more solute diffusion [McCormick, 1972a; Zhang et al., 2017]. Therefore, the waiting time of mobile dislocations influences the serration amplitude. Concerning the above mechanism, it is reasonable to link the different precipitate-dislocation interaction mechanisms to the serration amplitude, which will be discussed in the following.

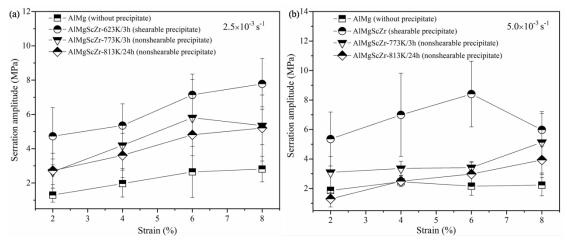


Fig. 7 Serration amplitude in AlMg, AlMgScZr-623K/3h, AlMgScZr-773K/3h and AlMgScZr-813K/24 alloys versus tensile strain at normal behavior (at higher strain rate): (a)  $2.5 \times 10^{-3}$  s<sup>-1</sup>; (b)  $5.0 \times 10^{-3}$  s<sup>-1</sup>. The serrations being analyzed are of type A+B serration.

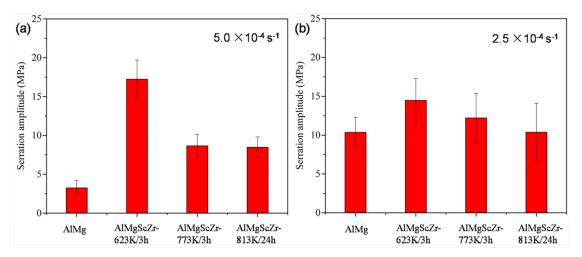


Fig. 8 Serration amplitude in AlMg, AlMgScZr-623K/3h, AlMgScZr-773K/3h and AlMgScZr-813K/24 alloys at same strain (10%) in inverse behavior: (a)  $5.0 \times 10^{-4}$  s<sup>-1</sup>, (b)  $2.5 \times 10^{-4}$  s<sup>-1</sup>. The serrations being analyzed are of type C serration.

## 4. Discussion

### 4.1 Strengthening effect induced by Al<sub>3</sub>(Sc, Zr) precipitates

It has been reported that the interaction between dislocation and precipitate changes from the shearing mechanism to the Oworan bypassing mechanism with the increase of precipitate size [Ardell, 1985; Gladman, 1999; Knipling et al., 2010; Ma et al., 2014; Ming et al., 2018]. When precipitate size is small, the dislocations can shear the precipitate, thus the dominant strengthening mechanism is the shearing mechanism. In general, the shearing mechanisms mainly include modulus strengthening, coherent

- strengthening and order strengthening [Ardell, 1985; Knipling et al., 2010; Ma et al.,
- 469 **2014**].
- Firstly, the modulus strengthening,  $\sigma_{\text{mod}}$ , due to the difference of shear modulus
- between precipitate and aluminum matrix, is given by [Ardell et al., 1985; Knipling et
- 472 al., 2010; Ma et al., 2014; Luca et al., 2019]:

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$$\sigma_{\text{mod}} = M \cdot 0.0055(\Delta G)^{3/2} (\phi / T)^{1/2} \cdot b(R / b)^{3m/2 - 1}$$
 (1)

- where M is Taylor factor, and M = 3.06 for face-centered cubic (FCC) polycrystal
- materials;  $\Delta G$  is difference on shear modulus between precipitates  $G_p$  ( $G_p=78$  GPa for
- Al<sub>3</sub>Sc precipitate) and aluminium matrix  $G_{\rm m}(G_{\rm m}=26.2~{\rm GPa}$  for aluminium matrix),
- 477 thus one has  $\Delta G = 41.8$  GPa in this study;  $\phi$  and R are the volume fraction and
- average radius of precipitates, which can be seen in Table 1; b is the Burgers vector,
- and b = 0.286 nm for aluminum; T is the line tension of dislocation, and  $T = 0.5Gb^2 =$
- 480  $1.072 \times 10^{-9}$  N; m is a constant, and m = 0.85.
- 481 Secondly, the coherent strengthening,  $\sigma_{\rm coh}$ , due to the elastic strain field
- interactions between coherent precipitates and dislocations, is given by [Ardell et al.,
- 483 1985; Knipling et al., 2010; Ma et al., 2014; Luca et al., 2019]:

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$$\sigma_{\text{coh}} = M \cdot \chi (\xi G_m)^{3/2} \cdot (R\phi b / T)^{1/2}$$
 (2)

- where  $\chi$  is a constant, and  $\chi = 2.6$ ;  $\xi$  is a misfit parameter, which describes the
- misfit degree between precipitate and dislocation, and  $\xi = 0.0179$ .
- Thirdly, the order strengthening,  $\sigma_{ord}$ , resulting from the formation of antiphase
- boundaries (APBs), occurs when the mobile dislocations shear the ordered precipitate,
- which is shown as [Ardell et al., 1985; Knipling et al., 2010; Ma et al., 2014; Luca et
- 490 al., 2019]:

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$$\sigma_{\text{ord}} = M \cdot (0.81 \eta_{APBs} / 2b) \cdot (3\pi\phi / 8)^{1/2}$$
 (3)

- where  $\eta_{APBs}$  is the average APBs energy for (111) plane, and  $\eta_{APBs} = 0.5 \text{ J m}^{-2}$  for
- 493 Al<sub>3</sub>Sc precipitate.
- When precipitate size is large, the dislocations cannot shear the precipitate, the
- 495 dominant strengthening mechanism is Orowan strengthening. The Orowan
- strengthening  $\sigma_{\text{oro}}$  can be expressed as [Knipling et al., 2010; Ma et al., 2014; Luca
- 497 et al., 2019]:

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$$\sigma_{\text{oro}} = 0.4MGb \ln \left(2\overline{R}/b\right)/\pi L_p \sqrt{1-\nu}$$
 (4)

where  $\overline{R}$  is the average planar radius, and  $\overline{R} = 0.25R$ ;  $L_p$  is the average 499 inter-distance of precipitates, and  $L_p = R\sqrt{(2\pi/3\phi) - \pi/2}$  . The values of  $L_p$  for 500 three AlMgScZr alloys are shown in Table 1. 501 For Al-Sc-Zr based alloy systems, the dominant precipitation strengthening 502 mechanism depends on the average radius of precipitate [Knipling et al., 2010; Lai et 503 al., 2013; Luca et al., 2019; Okle et al., 2019]. For smaller precipitates  $(R < R_1)$ 504 modulus plus coherency effects are dominant, while above  $R_1$  and below  $R_2$ , order 505 strengthening becomes important. If the precipitate size overcomes a certain limit 506  $(R > R_2)$ , order strengthening is replaced by the strengthening effect associated with 507 508 the Orowan bypassing mechanism. The increase in strength as a function of precipitate size can thus be summarized as follows: 509

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$$\Delta \sigma = \begin{cases} \sqrt{\sigma_{\text{mod}}^2 + \sigma_{\text{coh}}^2}, & R < R_1 \\ \sigma_{\text{ord}}, & R_1 < R < R_2 \\ \sigma_{\text{oro}}, & R > R_2 \end{cases}$$
 (5)

511 Here  $R_1$  is the critical radius between combined modulus-coherent strengthening and order strengthening;  $R_2$  is the critical radius between order strengthening (or shearing 512 513 mechanism) and Orowan strengthening. According to previous studies [Fazeli et al., 2008; Knipling et al., 2010; Okle et al., 2019; Chen et al., 2021], R₁≈1.2 nm and 514 R<sub>2</sub>≈3-4 nm for Al-Sc-Zr alloy [Fazeli et al., 2008; Knipling et al., 2010, 2011; Lai et 515 al., 2013; Luca et al., 2019; Okle et al., 2019]. Besides, we calculate the critical radius 516  $R_c$ , for the transition between the shearing and bypassing mechanisms, which 517 according to reference [Bréchet and Louchet, 1990]: 518

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$$R_c = \sqrt{3/2} (\Gamma/\gamma) \left[ 1 + 1.53 (\phi/\pi)^{1/2} \right]$$
 (6)

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where  $\Gamma$  is the line tension of dislocation;  $\gamma$  is the interface energy. It can be seen that  $R_c$  not only depends on the alloy system ( $\Gamma$ ) and precipitate type ( $\gamma$ ), but also depends on the volume fraction of precipitates. Generally, the value of  $\phi$  is much smaller than  $\pi$  for Al-Sc or Al-Sc-Zr alloys, thus  $R_c$  mainly depends on  $\Gamma$  and  $\gamma$ . According to Eq. (6), the calculated  $R_c = 2.8$  nm for Al<sub>3</sub>Sc precipitate (for (111) plane,  $\gamma = 0.5$  J m<sup>-2</sup> [Knipling et al., 2010]) and 3.1 nm (for the (111) plane,

 $\gamma = 0.445 \text{ J m}^{-2}$  [Lefebvre et al., 2014]) for Al<sub>3</sub>Zr precipitate. The calculated values 526 are close to those given by references [Fazeli et al., 2008; Knipling et al., 2010; Lai et 527 al., 2013; Okle et al., 2019], further supporting that the 3 nm is a reasonable value for 528 the critical size between shearing and bypassing mechanisms for Al-Sc-Zr alloy. 529 According to Eq. (5),the dominant strengthening mechanism in 530 AlMgScZr-623K/3h alloy is order strengthening, since the average size is 2.5 nm and 531 belongs to  $(R_1, R_2)$  range. Similarly, the dominant strengthening mechanisms in 532 AlMgScZr-773K/3h and AlMgScZr-813K/24h alloys are Orowan bypassing 533 mechanisms, since their average radius sizes (10.2 and 22.6 nm) are larger than  $R_2$ . 534 In order to verify that the determined strengthening mechanisms are reliable, the 535 results between experiments and strengthening model prediction are compared. Table 536 537 3 shows the yield stress of AlMg and three AlMgScZr alloys at different strain rates. The estimated yield stress increment as a function of precipitate size is shown in Fig. 538 9 together with the experimental values obtained in this work. For 539 AlMgScZr-623K/3h sample (Fig. 9(a)), the predicted values by combined 540 541 modulus-coherent strengthening, order strengthening and Orowan strengthening are 210, 159, 190 MPa, respectively. The experimental value is about 140 MPa, which is 542 543 close to the prediction of order strengthening. Thus, it is further verify that the dominant strengthening mechanism in AlMgScZr-623K/3h alloy is the order 544 strengthening. The experimental value is slightly smaller than the prediction of order 545 strengthening, which is due to the overestimated APBs energy of Al<sub>3</sub>Sc precipitate. 546 Our results are similar to that of Knipling et al. who reported that the calculated value 547 is higher than the experimental one due to the slightly overestimated APBs energy 548 [Knipling et al., 2010]. For AlMgScZr-773K/3h and AlMgScZr-813K/24h samples 549 (Figs. 9(b) and (c)), the predicted values by combined modulus-coherent 550 strengthening and order strengthening are much higher than experimental values. 551 While the predicted values by Orowan strengthening are very close to the 552 experimental values. Thus, it is further proved that the dominant strengthening 553 mechanisms are Orowan strengthening for these two samples. 554 In general, it can be confirmed that the interaction mechanisms between precipitate 555 556 and dislocation for three AlMgScZr alloys, providing the theoretical basis for analyzing the influence of precipitates on dislocation motion, distribution and 557 multiplication, and finally providing a good theoretical support for studying the 558

Table 3 Experimentally determined yield stress of AlMg ( $\sigma_{s1}$ ), AlMgScZr-623K/3h ( $\sigma_{s2}$ ), AlMgScZr-773K/3h ( $\sigma_{s3}$ ) and AlMgScZr-813K/24h ( $\sigma_{s4}$ ) alloys.

Strain rate (s <sup>-1</sup> )	1×10 <sup>-2</sup>	5×10 <sup>-3</sup>	1×10 <sup>-3</sup>	5×10 <sup>-4</sup>	1×10 <sup>-4</sup>
σ <sub>s1</sub> (MPa)	168.4±4.8	162.3±1.9	170.7±4.9	173.6±2.9	173.2±4.0
σ <sub>s2</sub> (MPa)	301.0±4.0	302.0±5.9	305.4±2.5	311.6±16.4	306.0±4.2
σ <sub>s3</sub> (MPa)	233.8±5.5	235.8±5.5	242.6±19.1	237.2±2.1	235.6±5.1
σ <sub>s4</sub> (MPa)	216.6±5.5	212.9±10.1	214.8±6.1	221.7±4.9	216.2±2.4

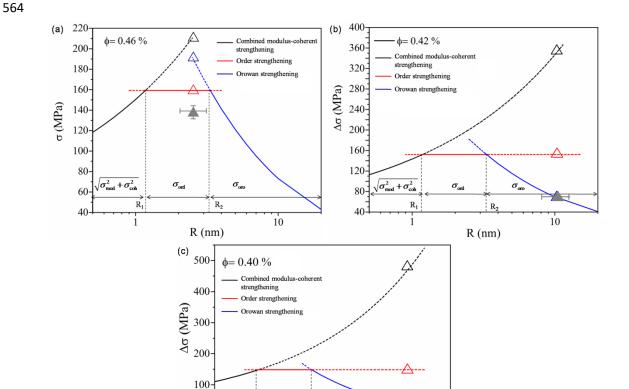


Fig. 9 Yield stress increment versus mean precipitate radius for (a) AlMgScZr-623K/3h [Chen et al., 2021], (b) AlMgScZr-773K/3h [Chen et al., 2021] and (c) AlMgScZr-813K/24h. The full curves in (a-c) represent calculated predictions of minimum of  $\sqrt{\sigma_{\rm mod}^2 + \sigma_{\rm coh}^2}$ ,  $\sigma_{\rm ord}$ , and  $\sigma_{\rm oro}$ . That is, it is taken the minimum value of  $\sqrt{\sigma_{\rm mod}^2 + \sigma_{\rm coh}^2}$ ,  $\sigma_{\rm ord}$ , and  $\sigma_{\rm oro}$  as the true strengthening increment according to

 $R_2$ 

R (nm)

 $\sigma_{\text{mod}}^2 + \sigma_{\text{coh}}^{2}$ 

the previous study [Knipling et al., 2010]. The solid symbols in (a-c) represent the yield stress increment obtained from tensile tests. The empty symbols in (a-c) refer to predictions for a certain precipitate radius and volume fraction obtained from SANS: (a) R = 2.5 nm,  $\phi = 0.46\%$ ; (b) R = 10.2 nm,  $\phi = 0.42\%$ ; (c) R = 22.6 nm,  $\phi = 0.40\%$ .

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#### 4.2 Effects of precipitates on dislocation motion, distribution and multiplication

The strengthening induced by Al<sub>3</sub>(Sc, Zr) precipitates is quantified in the 4.1 section, proving the different precipitate-dislocation mechanisms for different AlMgScZr samples in this study. According to Fig. 9, order strengthening dominates in the AlMgScZr-623K/3h alloy demonstrating that matrix dislocations shear ordered precipitates and create antiphase boundaries (APBs) on the slip plane in the precipitate. Moreover, Fig. 10 shows the precipitate structure of AlMgScZr-623K/3h alloy after a certain degree of tensile strain (5%). It can be evidently seen that the precipitates are sheared by dislocations in Fig. 10. The APBs energy per unit area can also be interpreted as the force per unit length opposing the dislocation motion when it penetrates the precipitate. During this process, dislocations travel in pairs (so called "superdislocations"), the second dislocation removes the APBs left by the first one [Ardell, 1985]. Recent work of Lefebvre et al. [Lefebvre et al., 2014] revealed that the APBs in Al<sub>3</sub>Zr can be the consequence of successive shearing of  $[\overline{1}10]/2$  Burgers vector dislocations gliding on distinct (111) planes. Under this circumstance, when the ahead dislocations pass through the precipitate, the subsequent dislocations are prone to move along the specific shear planes owing to its reduced motion resistance, leading to the formation of slip bands (Fig. 11). It can be seen from Fig. 11 that the slip bands with high density of dislocations form. It is clearly shown that lots of dislocation lines parallel with each other in the slip bands, indicating that dislocation pile-ups in slip bands are the results of dislocation motion along above specific shear planes. Although it is difficult to observe the Al<sub>3</sub>(Sc, Zr) precipitate inside the slip bands, the corresponding SAED of slip bands can confirm the existence of precipitates (Fig. 11(g)). Moreover, we observe the slip bands with relatively low dislocation density with the aid of scanning transmission electron microscope (STEM), as shown in Fig. A1 (supplementary materials). It observes lots of precipitates inside slip bands in the high magnification STEM images. Previous studies also have shown that the slip bands form due to dislocations shearing precipitates [Poole et al., 2005; Xiao et al., 2005; Sharma et al., 2009; Tayon et al.,

2019; Jaladurgam et al., 2020; Chen et al., 2021], which makes the distribution of dislocations extremely non-uniform. Due to the formation of slip bands, the strain is distributed in a limited volume for the AlMgScZr-623K/3h alloy with shearable precipitates. As a result, the local strain rate and the resultant local strain in the AlMgScZr-623K/3h alloy is higher than the macroscopic equivalent strain. Therefore, although single shearable precipitate cannot accumulate dislocations directly, the shearing process of such precipitates can store a large number of dislocations by the formation of slip bands.

With the shearable precipitates becoming nonshearable precipitates, the dislocation shearing mechanism is replaced by dislocation bypassing mechanism (for AlMgScZr-773K/3h and AlMgScZr-813K/24h samples). This process leaves Orowan loops around the nonshearable precipitates. The dislocations are distributed relative homogeneously during tensile deformation, which is different from that of AlMgScZr-623K/3h alloy with shearable precipitates, as shown in Figs. 12(b) and (c). It should be noted that the dislocations are also distributed uniformly for the AlMg sample without precipitates (Fig. 12(a)). Our results are similar to Poole et al. who reported that the shearable and nonshearable precipitates had distinct effects on dislocation distribution through different precipitate-dislocation interactions [Poole et al., 2005].

In order to quantitatively evaluate the dislocation density caused by precipitates, the dislocation densities of AlMg, AlMgScZr-623K/3h, AlMgScZr-773K/3h and AlMgScZr-813K/24h were evaluated by XLPA (the detailed evaluation procedure can be found in reference [Borbély and Groma, 2001]). The corresponding (200) peak profiles (as an example) and variations of dislocation densities versus tensile strain are shown in Fig. 13. The dislocation density of the AlMgScZr-623K/3h sample is larger by one order of magnitude compared with that of the AlMg alloy. Dislocation density decreases when shearable precipitates become nonshearable precipitates. The results are in very good agreement with the TEM images (Figs. 11 and 12). It should be noted that the AlMgScZr-623K/3h alloy has the highest dislocation density in all studied alloys, which is mainly due to its higher initial dislocation density retained during hot extrusion. Although the increment on dislocation density in

AlMgScZr-623K/3h alloy at the beginning stage of tensile deformation is higher than the others, the differences on dislocation density increment caused by tensile deformation for these four samples are relatively small. The AlMgScZr-623K/3h alloy with shearable precipitates has a higher dislocation density storage after hot extrusion deformation, which is mainly due to the fact that the shearable precipitates have stronger pinning effect on dislocations [Humphreys and Hatherly, 2004; Fazeli et al., 2008]. Thus, more dislocations can be retained during hot extrusion. The dislocations in AlMg alloy without precipitates cannot be arrested and pinned effectively by precipitates, thus the AlMg alloy has the lowest dislocation density storage after hot extrusion. Compared with AlMg alloy, the AlMgScZr-773K/3h AlMgScZr-813K/24 alloys have a higher density of dislocation storage due to pinning effect by the nonshearable precipitates, but their stored dislocation densities are smaller than that of AlMgScZr-623K/3h alloy, for their pinning effect on dislocations is weaker than that of shearable precipitates [Fazeli et al., 2008].



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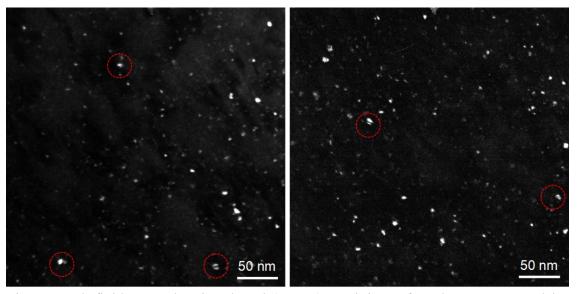
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Fig. 10 Dark-field TEM showing the Al<sub>3</sub>(Sc, Zr) precipitates for AlMgScZr-623K/3h alloy after 5% tensile strain. The zone axis is [011]. The dashed red circles clearly show that the precipitates are sheared by dislocations.

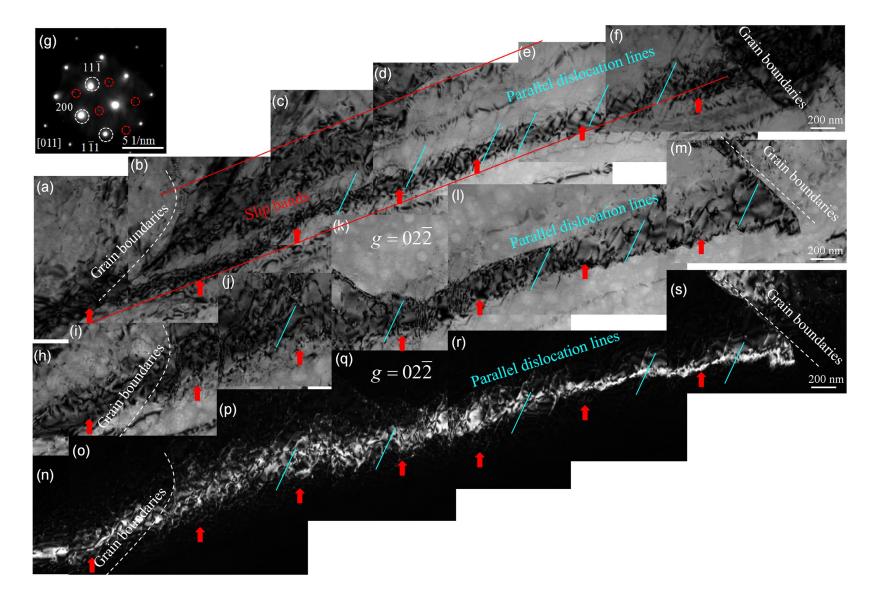


Fig. 11 TEM images of dislocation structure for AlMgScZr-623K/3h alloy with shearable precipitates after 5% tensile strain: (a-f) bright TEM images of slip bands with [011] zone axis; (g) is the corresponding SAED of (a-f); (h-m) bright TEM images of slip bands with dual beam diffraction ( $g=02\overline{2}$ ); (n-s) corresponding dark TEM images of (h-m) with dual beam diffraction ( $g=02\overline{2}$ ). Here the red full lines in (a-f) refer to the slip bands due to dislocation shearing precipitates. The slip bands labelled by red arrows in the (a-f), (g-l) and (m-s) are the same slip bands. The cyan dotted lines refer to the parallel dislocation lines, and the dashed white lines denote grain boundaries.

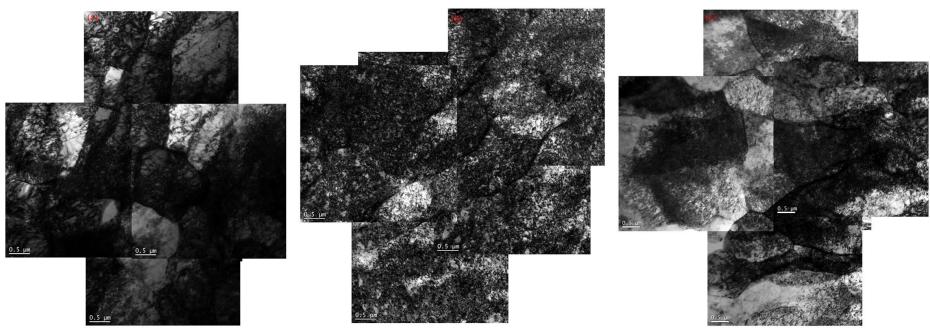


Fig. 12 Bright TEM images of dislocation structure for (a) AlMg (without precipitates), (b) AlMgScZr-773K/3h (with nonshearable precipitates) and (c) AlMgScZr-813K/24h (with nonshearable precipitates) alloys after 5% tensile strain. The TEM images of these three alloys are acquired with the [011] zone axis. The dislocations of above three alloys distribute relative uniformly compared with AlMgScZr-623K/3h alloy.

The TEM images of all samples after 5% tensile strain are presented to demonstrate the magnitude of dislocation density and dislocation distribution after tensile deformation (Figs. 11 and 12). For AlMgScZr-623K/3h alloy, after tensile deformation, lots of slip bands is observed in microstructure, and dislocations pile up inside these slip bands. It is reasonable to infer that the retained dislocations during hot extrusion can move along a specific direction to form slip bands due to the shearing mechanism between precipitates and dislocations (this process may also contain lots of new dislocations generated during tension). On the other hand, once the dislocations move and form slip bands, these dislocations are difficult to be annihilated during tension. This is because most of these dislocations inside slip bands have the same Burgers vector (most of dislocations lines are parallel, indicating that these dislocations are of same Burgers vector), thus they are difficult to be annihilated through cross-slip [Sandström, 1977; Nes, 1995; Xiao et al., 2005]. It should be noted that there are still a considerable number of dislocations outside the slip bands in AlMgScZr-623K/3h alloy, although its local density is much lower than that in slip bands. The measured dislocation density by SRXRD is total dislocation density, which contains the dislocations both inside slip bands and outside the slip bands. For AlMg and other AlMgScZr alloys, the dislocations distribute more uniformly (Fig. 12), these tangled dislocations usually have different Burgers vectors. Dislocation annihilation is easy to occur when dislocation tangles become complicated, which also limits the rapid increase of dislocation density [Verdier et al., 1998; Kocks and Mecking, 2003]. Thus, although alloys with nonshearable precipitates can accumulate lots of Orowan loops during tension, the total density of dislocations for a given microscopic tensile strain is much smaller (Fig. 13(f)) than in the case of shearable precipitates. The AlMgScZr-623K/3h alloy has the higher dislocation density than AlMg and the other AlMgScZr alloys is more related to the formation of slip bands (dislocations can be stored more effectively inside the local slip bands).

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The analysis in Figs. 11, 12 and 13 demonstrates that shearable and nonshearable

precipitates have different effects on dislocation storage during tensile deformation by changing dislocation motion and distribution. The change of dislocation distribution and density of stored dislocation for a given macroscopic tensile strain will affect the waiting time of dislocation in the localized bands and diffusion of solute atoms during DSA, thus inevitably influencing the propagating behavior, serration amplitude, onset strain and strain-rate range of PLC, which are discussed as follows.

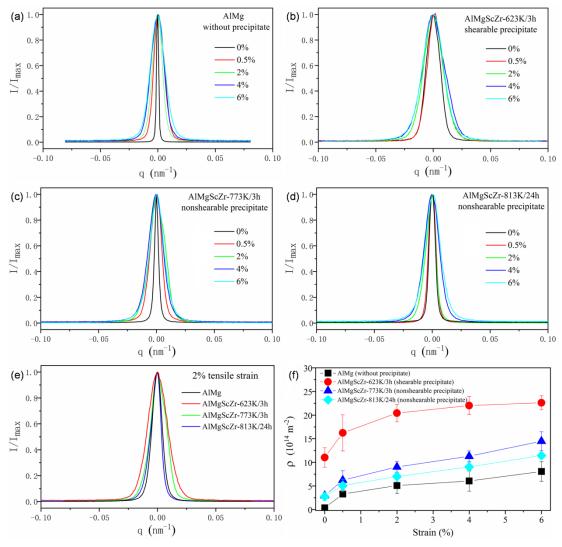


Fig. 13 Peaks profiles of AlMg and three AlMgScZr alloys as well as the quantitative measurement of dislocation density: (200) peaks profiles of (a) AlMg, (b) AlMgScZr-623K/3h, (c) AlMgScZr-773K/3h, and (d) AlMgScZr-813K/24h after different tensile strains; (e) (200) peaks profiles of AlMg and three AlMgScZr alloys after 2% tensile strain; (f) variations of dislocation density versus tensile strain for

AlMg and three AlMgScZr alloys. The dislocation density is measured averagely from (200), (220) and (311) peaks. Here the peaks profiles of (220) and (311) are omitted for simplicity, since they have similar features to the (200) peaks.

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## 4.3 Influence of precipitates on the characteristics of PLC bands

The PLC is due to the interaction between solute atoms and dislocations, which is the process dominated by dislocation pinning and unpinning [Kubin and Estrin, 1985; Rizzi and Hähner, 2004]. However, the solute-dislocation interaction mechanisms for the appearance of PLC are different in normal and inverse behavior regimes [Chaenock, 1969; Soler-gomez and Tegart, 1969; McCormick et al., 1970, 1972b; Fu et al., 2012; Zhemchuzhnikova et al., 2015; Zhang et al., 2017; Geng et al., 2020]. In normal behavior regime (at higher strain rate), the mobile dislocations usually have a higher movement velocity and the solute atoms cannot capture the mobile dislocations due to their relatively lower diffusion rate. When the mobile dislocations are arrested by forest dislocations and other obstacles, the solute atoms have the possibility to capture the mobile dislocations. Only when the solute atoms diffuse to the arrested mobile dislocations during the waiting time does PLC occur. Thus, at normal behavior, the PLC appears in the condition of the solute atoms diffusing to the arrested mobile dislocations and pinning these dislocations. The stress strain curves usually exhibit locking serrations or steps (Figs. 5(a), (c)), and the PLC appears in the form of an abrupt rise. The abrupt rise indicates a process that the solute atoms pin the arrested mobile dislocations, also meaning that the solute atoms pinning the mobile dislocations leads to PLC. Therefore, at this time, the physical mechanism for controlling the appearance of PLC is defined as "dislocation pinning mechanism". On the contrary, in inverse behavior regime (at lower strain rate), the mobile dislocations can be pinned by solute atom atmosphere before the tensile plastic deformation due to the very low velocity of mobile dislocations. The critical condition for the appearance of PLC is the breakaway of mobile dislocations from this atmosphere, which is different from that in normal behavior regime [Chaenock, 1969; Soler-gomez and Tegart, 1969; McCormick et al., 1970, 1972b; Fu et al., 2012; Geng et al., 2020]. The stress strain curves usually manifest unlocking serrations (Fig. 5(f)) for inverse behavior, and the PLC appears in the form of a stress drop. The stress drop indicates a process that the mobile dislocations escape from the pinning of solute atmosphere,

meaning that the escape of mobile dislocations from the existing atmosphere also leads to the PLC. The physical mechanism controlling the appearance of PLC is thus defined as "dislocation unpinning mechanism".

It is shown that precipitates influence the transition between A+B/B and C type bands: the corresponding strain rate firstly increases from  $5.0 \times 10^{-4}$  s<sup>-1</sup> to  $2.5 \times 10^{-3}$  s<sup>-1</sup> for the AlMgScZr-623K/3h alloy with shearable precipitates followed by a decrease from  $2.5 \times 10^{-3}$  s<sup>-1</sup> to  $7.5 \times 10^{-4}$  s<sup>-1</sup> when shearable precipitates become nonshearable precipitates (Figs. 6(a-d)). These results show that precipitates increase the lower strain rate for the occurrence of type C band. Our results are similar to Cai et al. who manifested that precipitates increase the lower strain rate for the appearance of type C band in Ni-based superalloys [Cai et al., 2015, 2017]. Regarding as sites where the dislocations pile up (Fig. 11), shearable precipitates increase the internal stress and favor a change from type B band to type C band with the decrease of strain rate (Figs. 6(a-d)). The shearable precipitates strongly influence the dynamical mechanisms of local strain propagation, through the localization of dislocation glide (Fig. 11) [Mogucheva et al., 2016].

The type C bands are a kind of dislocation unlocking serrations due to the unpinning mechanism between mobile dislocation and solute atoms, while type A and B bands are caused by dislocation pinning mechanism. Shearable precipitates increase the lower strain rate for the formation of type C band (Fig. 6(a-d)), meaning that the shearable precipitates increase the critical strain rate between dislocation unpinning and pinning mechanisms. It is because that, at same strain rate, the high local dislocation density in alloy containing shearable precipitates could enhance the diffusion rate of solute atoms (Fig. 13(f)). On one hand, the higher density of dislocation can reduce the diffusion activation energy and thus enhances the diffusion velocity [Legros et al., 2008]. On the other hand, the substitutional solute migration is correlated with the vacancy concentration [Picu and Zhang, 2004; Picu and Xu, 2007; Van der Ven et al., 2010]. As such systems with larger dislocation content are more likely to exhibit local vacancy supersaturation which will aid diffusion [Van der Ven et al., 2010]. When the diffusion rate of solute atoms increases, it is much easier to meet the condition of dislocation unpinning (i.e. the diffusion rate of solute atoms is much higher than the velocity of mobile dislocations). At this time, there is no need to decrease the movement velocity of mobile dislocation to meet dislocation unpinning via decreasing the strain rate. Therefore, the shearable precipitates increase the critical

strain rate between unpinning and pinning mechanisms by increasing local dislocation density for a given microscopic strain. However, these effects (increasing the lower strain rate for the formation of type C band and increasing the critical strain rate between unpinning and pinning mechanisms) weakens due to the decrease of stored dislocation density when shearable precipitates become nonshearable precipitates (Figs. 6(a-d) and Table 2).

As far as the serration amplitude is concerned, the appearance of precipitates increases the magnitude of serration amplitude no matter for normal behavior (Fig. 7) or inverse behavior (Fig. 8). The serration amplitude  $\Delta \sigma$  can be expressed as [Chen et al., 1996; Zhang et al., 2017]:

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$$\Delta \sigma \propto \left[ \dot{\varepsilon}^{-1} \varepsilon^{\beta(1/2+\gamma)} d^{-n(1/2+\gamma)} T^{-1} \exp(-Q/kT) \right]^{2/3}$$
 (7)

where  $\dot{\varepsilon}$  is strain rate;  $\varepsilon$  is strain; d is the grain size; T is temperature; Q is the activation energy for solute diffusion, which is associated with solute-vacancy site exchange; k is the Boltzmann constant. Precipitates enhance the accumulation of dislocation density, which can reduce Q and thus accelerate the diffusion of atoms [Legros et al., 2008]. Therefore, it is reasonable to infer that the  $\Delta \sigma$  increases for AlMgScZr alloy with precipitates in contrast to AlMg alloy based on Eq. (7). By comparing the samples with shearable and nonshearable precipitates, the shearable precipitates can reduce Q obviously by storing more dislocations, leading to a larger serration amplitude.

#### 4.4 Influence of precipitates on normal and inverse behaviors

Since the solute-dislocation interactions for the appearance of PLC at normal and inverse behaviors are related to the dislocation pinning and unpinning mechanisms, respectively [Chaenock, 1969; Soler-gomez and Tegart, 1969; McCormick et al., 1970, 1972b; Fu et al., 2012; Geng et al., 2020]. The introduction of shearable precipitates and nonshearable precipitates have different effects on different solute-dislocation interactions, which are discussed as follows.

#### 4.4.1 Influence of precipitates on normal behavior

At higher strain rates, the locking serration (type A and B bands) or upward step

(type A bands) demonstrates that mobile dislocations are not pinned by diffusing solute atoms at the beginning of tensile deformation [Chaenock, 1969; Soler-gomez and Tegart, 1969; McCormick et al., 1970, 1972b; Fu et al., 2012; Geng et al., 2020]. When the mobile dislocations are captured by diffusing solute atoms, the PLC occurs. For the AlMg alloy mobile dislocations are mainly pinned by forest dislocations, while in AlMgScZr the mobile dislocations are pinned both by forest dislocations and precipitates together. When the shearable precipitates are present, the mobile dislocations will be additionally pinned until they can find a partner dislocation to form the superdislocations and cut through the precipitates. Thus, the pinning effect on mobile dislocations increases evidently when the shearable precipitates appear. As a consequence, the waiting time  $t_w$  also increases obviously. When shearable precipitates become nonshearable, the mobile dislocations will be additionally pinned until they pass through the precipitates. Compared with the shearable precipitates, the pinning effect on mobile dislocations provided by nonshearable precipitates weakens (Fig. 9 and Table 3). Thus, the waiting time  $t_w$  decreases with the shearable precipitates transforming to nonshearable. Here  $t_w$  refers to the time during which the mobile dislocations are arrested by obstacles, which is given by Bréchet and Estrin, 1995; Curtin et al., 2006]:

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$$t_{w} = \Omega / \dot{\varepsilon} = \rho_{m} b L / \dot{\varepsilon}$$
 (8)

where  $\Omega$  is elementary plastic strain [Bréchet and Estrin, 1995];  $\rho_m$  is denoted as mobile dislocations; b is Burgers vector; L is the average spacing between obstacles, representing the flight distance of solute atom migration;  $\dot{\varepsilon}$  is the strain rate. Compared to the AlMg alloy, the waiting time  $t_w$  of mobile dislocations in AlMgScZr alloy with shearable precipitates increases, which indicates that the solute atoms have more time to capture the arrested mobile dislocations. At this time, there is no need to enhance the diffusion velocity of solute atoms by imposing a higher tensile strain, and thus the onset strain of PLC decreases. When the shearable precipitates transform into nonshearable precipitates, the waiting time  $t_w$  decreases, and the solute atoms need a higher diffusion velocity in order to capture the arrested mobile dislocations. At this time, a relative higher tensile strain is required for generating more vacancies to enhance diffusion velocity of solute atoms, thus the onset strain increases. In summary, the presence of shearable precipitates and their

subsequent transforming into nonshearable precipitates make the imposed onset strain to meet the pinning requirement first decreased and then increased at high strain rates, as shown in Figs. 6(e-h).

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## 4.4.2 Influence of precipitates on inverse behavior

At lower strain rates, the unlocking serration (type C band) indicates that mobile dislocations are pinned before tensile plastic deformation. When the mobile dislocations escape from pinning obstacles, the PLC occurs [Chaenock, 1969; Soler-gomez and Tegart, 1969; McCormick et al., 1970, 1972b; Fu et al., 2012; Geng et al., 2020]. Thus the PLC phenomenon will occur after a certain degree of tensile plastic deformation. In the process of mobile dislocations overcoming obstacles, the mobile dislocations need to overcome the energy barrier. It is obvious that the energy barrier will increase due to the introduction of precipitates and the strain rate for the onset of PLC will increase correspondingly. The results in sections 4.1 and 4.2 have proved that the shearable precipitates improve the strength and store a higher density of dislocation density, indicating that the applied stress will be increased if the mobile dislocations expect to overcome the energy barriers. However, the inhibition effects decrease due to the shearable precipitates becoming nonshearable precipitates, and as a result the energy barrier will be decreased due to the weaker precipitate-dislocation interaction (the subsequent decrease of strength and dislocation density can prove it (Figs. 9 and 13)). Hence, the shearable precipitates will make the low critical strain rate for the occurrence of PLC increased evidently, and this low critical strain rate will decrease with the shearable precipitates being replaced by nonshearable precipitates. This explains that the shearable precipitates firstly narrow the strain-rate ranges for the appearance of PLC and then expands this strain-rate range with the shearable precipitates becoming nonshearable (Fig. 6(a-d) and Table 2). For the onset strain, if the strain rate is same, the first increase and then decrease of energy barriers by introducing shearable precipitates and subsequent growing into nonshearable precipitates result in the corresponding variations of onset strain. It is because dislocations can escape the higher energy barrier only by increasing onset strain to provide a higher stress. Therefore, at inverse behavior regime, the shearable precipitates first enlarge the critical strain for the appearance of PLC and then decreases this strain when shearable precipitates become nonshearable precipitates

870 (Figs. 6(i-1)).

It should be noted that the process of dislocation shearing small precipitates is influenced by thermal activation for AlMgScZr-623K/3h alloy according to Argon's study [Argon, 2008]. At higher strain rate, the mobile dislocations have a higher movement velocity, which is easier to cutting the precipitates and overcome the energy barrier, since the mobile dislocations have a higher kinetic energy. Thus, at this time, the dislocations have a higher cutting rate, and the waiting time provided by precipitates will decrease to some extent.

#### 5. Conclusions

In this study, we systematically investigate the influence of shearable and nonshearable Al<sub>3</sub>(Sc, Zr) precipitates on PLC behavior for precipitation hardening AlMgScZr alloys. It is shown that the presence of precipitates, as well as the change of the precipitate-dislocation interaction mechanism leads to different PLC behavior. The main conclusions are the following:

(1) Shearable precipitates promote the formation of slip bands with dislocation pile-ups due to their precipitate-dislocation interaction, while the dislocations distribute relatively uniformly in samples containing nonshearable precipitates. Compared with the samples with nonshearable precipitates, the higher local strain rate and higher local strain caused by shearable precipitates make more dislocations retained in samples, leading to a higher dislocation storage.

(2) Shearable precipitates increase the lower strain rate for the appearance of type C band, but this effect decreases with the shearable precipitates becoming nonshearable precipitates. Shearable precipitates enhance the serration amplitude largely, but the serration amplitude decreases when shearable precipitates become nonshearable precipitates. Both above phenomena are attributed to the first increase and then decrease of dislocation density controlled by different dislocation-precipitate interactions.

(3) At higher strain rates, the shearable precipitates decrease the onset strain but the onset strain then increases when shearable precipitates become nonshearable

precipitates, which is due to the first increase and then decrease of waiting time controlled by different precipitates. At lower strain rates, the shearable precipitates increase the onset strain but the onset strain then decreases with the shearable precipitates being replaced by nonshearable precipitates, which is due to the first increase and then decrease of energy barrier which needs to be overcome.

(4) Shearable precipitates narrow the strain-rate range of PLC, but this strain-rate range expands again when shearable precipitates are replaced by nonshearable precipitates. That is because the lower critical strain rate being dominated by the precipitate-induced energy barrier first increases and then decreases.

(5) At higher strain rates, precipitates act as additional obstacles, along with forest dislocations, to arrest mobile dislocations. Solute atoms thus have more time to diffuse onto arrested mobile dislocations, leading to a decrease of onset strain, i.e., precipitates facilitate dislocation pinning process. At lower strain rate, additional pinning effect provided by precipitates results in a higher required applied stress for arrested mobile dislocations to escape from the pinning of atmosphere, leading to an increase of onset strain, i.e., precipitates prevent dislocation unpinning process. Both of the above effects attenuate with the shearable precipitates becoming nonshearable precipitates.

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