

Investigation Of Shear Band Formation For In-Situ Magnesium Metal Matrix Composites During Hot Compression

Nagaraj M. Chelliah¹, Gopalakrishnan P.², and M.K. Surappa³

¹Department of Metallurgical Engineering PSG College of Technology Coimbatore
641004, Tamil Nadu, INDIA

^{2,3}Department of Materials Engineering Indian Institute of Science Bengaluru 560012,
Karnataka, INDIA

Email: ¹nag.metal@psgtech.ac.in

Abstract

Twinning and dynamic recrystallization are believed to be the two major contributors for shear band formation in polycrystalline magnesium. In this present work, we have proposed an empirical relationship to determine the criteria for shear band formation based on work-hardening values derived from true stress-true strain curves. In-situ magnesium metal matrix composites were subjected to hot compression tests at strain rate ranges of 1×10^{-3} to 1 s^{-1} and from temperature ranges of 150 to 350 °C. We show that if the ratio of work hardening or work-softening rate to maximum work hardening rate reduces below -0.02, then the shear bands nucleate within the grain matrix and propagate along the direction having maximum shear stress. On the other hand, if this ratio exceeds 0, no shear band formation occurs within the composites. However, when temperature approaches 350 °C, activation of non-basal slip systems suppresses the severity of twinning by enhancing the plastic flow across the several grains, which eventually leading to produce deformation bands rather than shear bands.

Keywords: Magnesium; Metallic Composites; Microstructure; Twinning; Shear band; Work hardening

1. INTRODUCTION

In recent years, in-situ magnesium metal matrix composites appear to be gaining interest among the several researchers for their potential applications in aerospace, automobiles and defense industries because of enhanced interfacial shear strength between ceramic/metal phases [1-3]. Investigators at the Indian Institute of Science and the University of Colorado have been collaborating to explore the possibility of enhancing the high temperature creep performance in polymer-derived in-situ magnesium metal matrix composites (P-MMCs) by utilizing the in-situ pyrolysis approach [4-5]. Owing to the existence of limited slip systems in hexagonal closed packed (HCP) crystal, magnesium tend to deform preferably by twinning mode at room temperature. However, magnesium deforms by dislocation assisted slip once the activation of slip systems occurs at high temperature regimes. Partridge [6] discussed that the deformation modes and operation of slip systems (combination of slip planes and slip direction) in magnesium are determined by four important factors namely: (i) Von-Mises

criterion, (ii) Schmid factors, (iii) critical resolved shear stress (CRSS), and (iv) temperature dependence of CRSS. Li et al. [7] examined the influence of grain size on the transition between twinning and slip assisted deformation modes and were able to determine quantitatively the critical grain size as 2 μm below which dislocation mediated slip dominates in Mg polycrystals. Chapuis et al. [8] studied the temperature dependency of slip and twinning in Mg single crystals by performing plain strain compression tests from ambient temperature to 450 $^{\circ}\text{C}$. They concluded that critical CRSS of basal slip and tensile twinning planes are not much temperature dependent whereas CRSS of prismatic, pyramidal, and compression twinning planes decreases significantly by a factor of three as temperature increases from 150 $^{\circ}\text{C}$ to 350 $^{\circ}\text{C}$.

Shear bands are highly localized strain regions that can transverse across several grains leading to pre-mature failure in magnesium and its alloys during deformation. The fundamental understanding of SB development in MMCs is of significant interest among the researchers as it provides the framework for indentifying an un-safe processing domain during metal forming operation. However, the underlying mechanisms behind the formation of shear bands in magnesium are still under several controversies [9-10]. Twinning and dynamic recrystallization are believed to be the two important sources for nucleating shear bands in magnesium. Sandlobes et al. [9] believed that shear band is formed within the matrix as a result of localized strain accumulation imposed by twinning. CT and double twins (DT) have a stronger tendency to deform solely by basal plane owing to softer orientation for basal slip than the matrix. As deformation progresses, strains are being accumulated and concentrated within this twin area which eventually leading to develop SB. On the contrary, Ion et al. [10] proposed that SB formation occurs in a material owing to dynamic recrystallization (DRX) around the grain boundaries (GB). Recrystallized grains with necklace arrangement have a softer orientation for basal slip leading to accumulate localized strains, and SB. The SB might form at 45 $^{\circ}$ with respect to the compression axis owing to maximum shear stress direction. Selvam et al. [11] investigated the effect of strain rate and temperature on compression behavior of extruded Mg matrix nanocomposites and they observed that the tendency of adiabatic shear band formation increases with increasing strain rate and temperature due to inadequate time available for the transfer of heat during plastic deformation.

Prasad et al. [14] studied the hot workability and deformation behavior of magnesium metal matrix nanocomposites containing nano-alumina particles in the temperature ranges of 300-500 $^{\circ}\text{C}$ and strain rate range of 3×10^{-4} - 10 s^{-1} . By generating processing maps, Prasad et al. [14] found that optimum condition for hot working of Mg matrix nanocomposite occurs at a temperature of about 450 $^{\circ}\text{C}$ and 10 s^{-1} whereas instability regime occurs in the temperature range of 400-500 $^{\circ}\text{C}$ and at strain rate below $1 \times 10^{-2} \text{ s}^{-1}$. Despite the fact that numerous data are reported for shear band formation and deformation mechanisms in various metal matrix composites [15-18], the clear insight regarding shear band development in in-situ magnesium metal matrix composites is yet to be explored. Main objective of the present manuscript was to reveal the underlining mechanisms for the development of shear bands in in-situ magnesium MMCs containing polymer derived SiCNO dispersoids at temperature (150-350 $^{\circ}\text{C}$) and strain rate ranges (1×10^{-3} - 1 s^{-1}) under uni-axial compression.

2. EXPERIMENTAL PROCEDURES

2.1 Materials And Methods

Commercially grade Mg (99.9%) billets (Supplied by Hindustan Aerospace Limited, Bengaluru) was chosen as the matrix material. The chemical composition of pure Mg is given in Table 1. Poly(urea-methyl-vinyl)silazane(PUVMS) procured from Kion Corporation (USA), was utilized as polymer precursor for the reinforcement. In-situ MMCs were fabricated by injecting a liquid polymer into the molten magnesium at 700 °C using liquid stir-casting method. The projected volume fraction of reinforced polymer derived SiCNO ceramic particles in the magnesium matrix composite is about 0.25. The detailed procedures of fabrication process had been discussed in Ref [19].

2.2 Hot Compression Tests And Microstructural Analysis

Cylindrical specimens having 6.5 mm diameter and 12 mm length were machined out of the as-synthesized composites. The flat end of the compression specimens had grooves of 0.1 mm depth in order to provide lubrication during compression. Corners of the compression specimens were chamfered by 0.5 mm to avoid the effect of fold and barreling during initial stage of compression. Isothermal uniaxial compression tests were performed on Dartec mechanical testing machine at constant true strain rates ranging from 10^{-3} to 1 s^{-1} for the temperature range of 423–623 K (150 to 350 °C). All the specimens were deformed up to half of their initial length corresponding to true strain of 0.69 and then quenched in oil to preserve the deformed microstructures. The load-displacement data were transformed into true stress-true strain curves using standard equations. The deformed specimens were sectioned along their compression axis, cold mounted, mechanically polished, and then chemically etched by using the standard metallographic procedures. The microstructures in both as-cast and compressed in-situ composite specimens were observed by scanning electron microscope (JEOL JSM-6610LV, Japan) and optical microscope (Leica DM2700, Germany).

3. RESULTS AND DISCUSSION

SEM micrographs of as-cast composites containing polymer derived SiCNO ceramic dispersoids are shown in Fig.1. The microstructures were characterized by segregation of the SiCNO dispersoids at vicinity of the grain boundaries as marked by several arrows in Fig.1(a) and Fig.1(b).

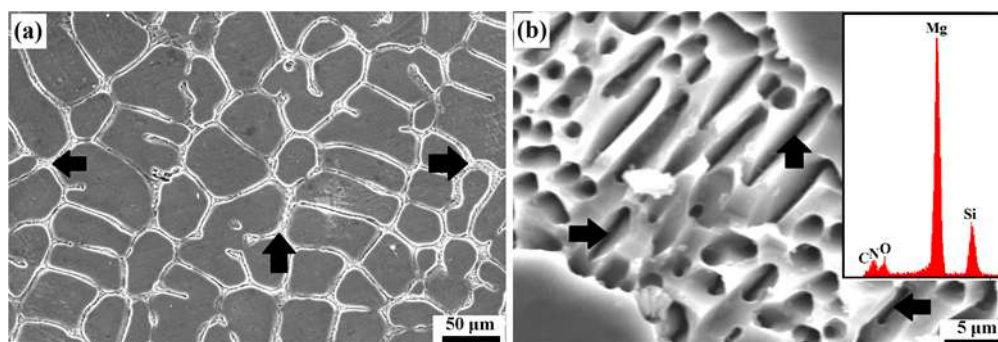
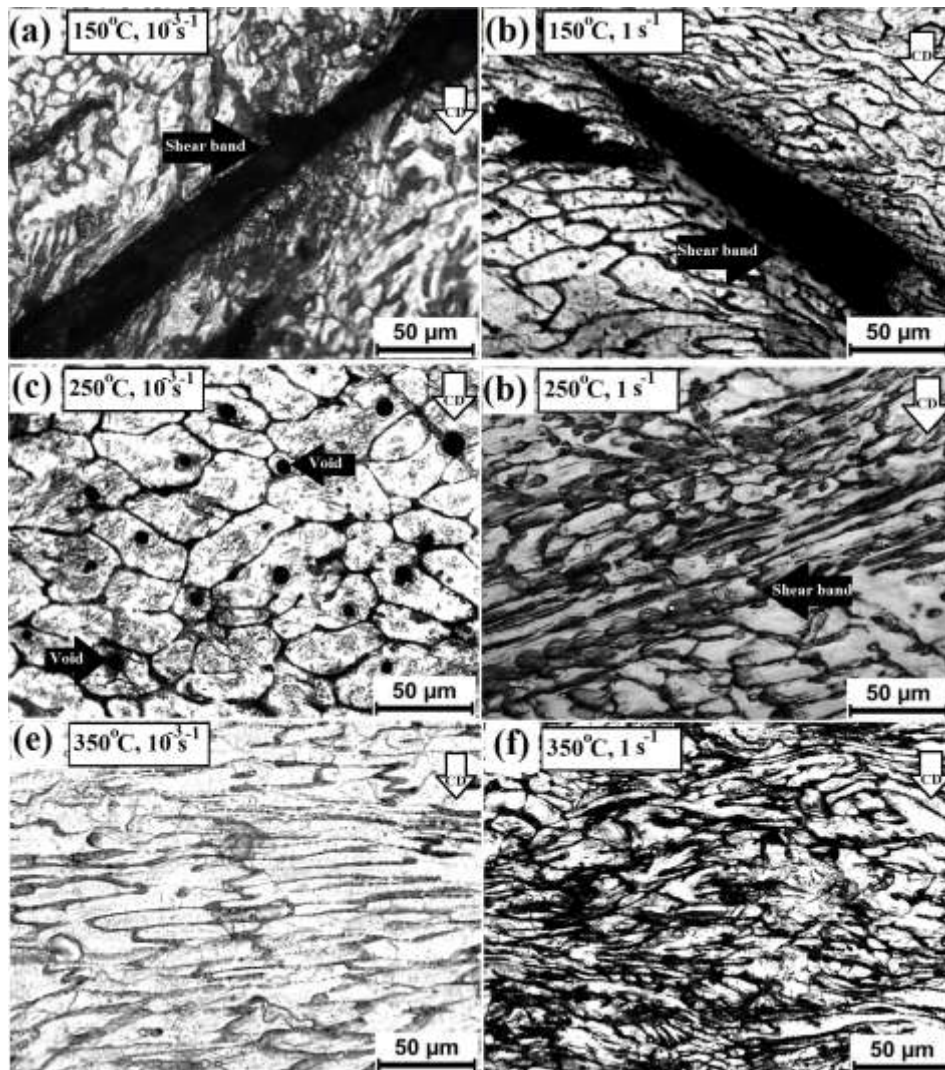


Fig: 1 Microstructures of as-cast Mg-based composites containing SiCN particles

The primary reason behind this segregation is that most of the SiCNO dispersoids are rejected and pushed away by the solidification front during casting process. The typical grain size of as-cast composites is estimated to be in the range of 50-65 μm . As shown in Fig 1(b), it can be also seen that the SiCNO dispersoids had a platelet morphologies having a width of 0.5-1

μm and length of 3-5 μm . As seen in Fig. 1(b), EDAX spectrum confirms the presence of Si, C, N and O elements for the polymer derived ceramic dispersoids. Macrographs of the as-cast in situ Mg matrix composites deformed at different strain rates and temperatures are shown in Fig. 2. Fig. 3 shows the microstructures of the composites deformed at different temperatures (150 °C, 250 °C and 350 °C) for the constant strain rate of 10^{-3} and 1 s^{-1} .



The compression direction (CD) is indicated by white arrow in all the optical micrographs. At 150 °C, both the strain rate conditions lead to the formation of shear band oriented at an angle of about $41-45^\circ \pm 6^\circ$ with respect to compression axis as indicated by arrow in Fig. 3(a) and Fig. 3 (b). At 250 °C with a lower strain rate of 10^{-3} s^{-1} , several voids are observed within the matrix of in-situ composites as depicted by arrows in Fig 3(c). These voids may form as a result of localized vacancy diffusion, and almost stabilized inside the grain itself. As observed in Fig. 3(d), grains are almost oriented at an angle of around $42-45^\circ$ with regard to compression axis when the strain rate is increased to of 1 s^{-1} at 250 °C . This could be associated with twinning activity along the direction of maximum shear stress due to the limited slip systems available for plastic deformation. This difference in microstructures is mainly occurred due to change in the strain rate during hot compression. For instance, lower

strain rate condition (10^{-3} s^{-1}) provides enough time available for vacancy diffusion to form voids whereas higher strain rate (1 s^{-1}) condition tends to produce twinning in the magnesium composites. The average width of shear band is estimated to be in the range of 55 to $5 \pm 35 \mu\text{m}$ for all the composites tested at 150 and $250 \text{ }^\circ\text{C}$. At $350 \text{ }^\circ\text{C}$, the composite microstructures are characterized by the presence of elongated grains irrespective of strain rates as shown in Fig 3(e) and Fig 3(f). While lower strain rate condition produces elongated grain structures which are oriented in a direction perpendicular to the compression axis, the higher strain rate condition develops elongated grain structures distributed in the form of wavy patterns. This demonstrates that plastic deformation is more or less homogenous at lower strain rate condition and it becomes non-homogenous in nature for the case of higher strain rate condition at $350 \text{ }^\circ\text{C}$.

The flow curves of the as-cast composites and their corresponding work-hardening response (θ) at strain rate range (1×10^{-3} - 1 s^{-1}) for different temperatures are shown in Fig. 4. It is evident that the flow curves of the composites deformed at $150 \text{ }^\circ\text{C}$ exhibit concave characteristics irrespective of strain rate ranges (Fig. 4(a) and Fig. 4(c)). The existence of concave region is a clear signature for the onset of extension twinning. Such a point of view has been reported by several researchers in Refs [5-6]. Interestingly, the concave nature is also followed by additional strain-hardening before it achieves strain of about 0.65 as shown in Fig 4(c). This additional work-hardening may be recognized as a manifestation of strong interactions between dislocation-twin grains as well as dislocation-SB within the composites during subsequent deformation. It should be kept in mind that room temperature compression always tends to develop shear band formation owing to the activation of twinning phenomenon if basal planes are oriented perpendicular to the compression direction.

As shown in Fig 4(b) and Fig 4(d), the work-hardening response of the deformed composites can be distinguished into two regimes based on the value of θ , viz: (i) work-hardening regime ($\theta > 0$), and work-softening regime ($\theta < 0$). At lower strain range (0 to 0.05), work-hardening rates of the composites suddenly drop down on yielding due to elastic-plastic transition. Subsequently, θ remains more or less constant for a short period and then steadily decreases until it reaches minimum. At this moment, it is worthwhile to consider the geometrical model for shear band formation. The evolution of shear band in magnesium under plain strain compression is explained by Sandlobes [9] as follows; (i) basal planes are oriented perpendicular to the compression axis and hence basal slip cannot accommodate any compressive strain (ii) This scenario activates compression twinning or double twinning and thereafter the basal planes within the twin area are preferably oriented for basal slip, (iii) The localized strain accumulation within the twin area creates several fine twins which eventually tends to nucleate shear band. It is to be noted that all the compression specimens are machined from the as-cast composites in such a way that basal planes are oriented perpendicular to the direction of compression. Therefore, there exists a possibility of activating the compression or double twins which results in reorientation of basal planes by about 58° and 38° respectively from their original positions during hot compression. These twins induce preferential orientation and promote basal slip activity in the in-situ composites. The presence of deep valley in work-softening regime at $150 \text{ }^\circ\text{C}$ represents the formation of shear band (see Fig 4(b) and Fig 4(d)). Couling et al. [21] recognized that the shear bands are certainly “soft” in nature aligning the easy glide basal planes more favorably for shear within the banded material. Dai et al. [22] reported that high strain gradient also provides a driving force for the formation of shear band in the MMCs. Notice that most of the SiCNO dispersoids are segregated at the vicinity of grain boundaries in the as-cast composites. Therefore, the possibility of achieving high strain gradient enhances within the single grain

itself. This tends to develop shear band formation within the composites. A noteworthy feature of the work-hardening curve is that if the ratio of rates of work-softening or work-hardening to maximum work-hardening reduces below -0.02, then shear band can be propagated along the direction of maximum shear stress as shown in Fig. 4(e). On the other hand, no shear band formation occurs if ratio of rates of work-softening or work-hardening to maximum work-hardening approaches above 0 as indicated in Fig 4(f). We have proposed a novel criterion for shear band formation purely based on semi-quantitative approach (i.e., the ratio of observed work-softening or work-hardening rate to the maximum work-hardening rate from the derivative of true stress-true strain curves obtained during hot compression but not any rigorous mathematical derivation. Therefore, we can invoke a mathematical relation for shear band formation as follows:

(i) Shear band formation occurs if

$$\frac{\text{Observed work – hardening or work – softening rate}}{\text{Maximum work – hardening rate}} = \frac{\left[\frac{d\sigma}{d\varepsilon}\right]}{\left[\frac{d\sigma}{d\varepsilon_{max}}\right]} \leq -0.02$$

(ii) No shear band formation occurs if

$$\frac{\text{Observed work – hardening or work – softening rate}}{\text{Maximum work – hardening rate}} = \frac{\left[\frac{d\sigma}{d\varepsilon}\right]}{\left[\frac{d\sigma}{d\varepsilon_{max}}\right]} \geq 0$$

The physical basis of this critical ratio from the point view of strain energy is not clear to us, presently. Nevertheless, the strain at which shear band occur at 150 °C is typically in the range of 0.28-0.40. However, this strain was increased to 0.57 at 250 °C. Further, there is no evidence of work-softening regime in the composites at 250 °C except at strain rate of 1 s⁻¹. On the contrary, when temperature approaches 350 °C, activation of non-basal slip systems suppresses the severity of twinning by enhancing the plastic flow stretch across the several grains, which eventually leading to produce elongated grain structures in the composites.

Microstructural analysis revealed that composites exhibit no dynamic recrystallization under any temperature and strain rate ranges in Fig 3(a) through Fig 3(f). As shown in Fig 1(b), it is observed that triple junction of grain boundaries are occupied with thermally stable SiCNO dispersoids. This suggest us that grain boundary do not slide, rotate, or migrate but it allows grains only to elongate after compressive deformation as shown in Fig 3(e). In fact, Prasad et al [14] reported that DRX does not takes place if prior particle boundaries are decorated by nano-alumina particles in magnesium matrix nanocomposites during compressive deformation. Moreover, Zhou et al [23] pointed out that true stress-strain curves of AZ91 magnesium matrix composites exhibit specific characteristic associated with DRX; i.e rapid increase in flow stress up to point of peak flow stress during initial stage of compression, and then the composite experience decrease in flow stress until it achieves steady state as the deformation continues. However, there is no such signature of steady state flow stress is recorded in any of the true stress-strain curves ((Fig 4(a) & Fig 4(b)). This means that rate of work hardening is not balanced with rate of work softening during hot compression of in-situ magnesium composite at any combination of temperatures and strain rates. Therefore, the role of dynamic recrystallization on shear band formation could be ruled out in the present work.

4. CONCLUSIONS

We have proposed a novel criterion for the formation of shear bands in in-situ MMCs based on hot compression measurements. We show that ratio of work-softening or work-hardening/maximum work-hardening rates, twinning tendency and high strain gradient are the three primary contributors that encourage the formation of shear bands in the in-situ magnesium composite during hot compression. We also provide a convincing proof regarding the development of shear bands from microstructural evidences as well as work-hardening curves.

Acknowledgement

The authors would like to express their gratitude to Prof. Rishi Raj, University of Colorado, Boulder, for providing the precursor materials under Grant No.DMR1105347 supported by the National Science Foundation. The authors would also like express their gratitude to Mr. K. Raja and Mr. S. Sasidhara, Indian Institute of Science, Bengaluru for their extensive help in the composite machining and in conducting the hot compression tests, respectively.

5. REFERENCES

- [1] S.C. Tjong, and Z.Y. Ma, *Mater. Sci. Engg: R.* 29, 49 (2000).
- [2] B.L. Mordike, T. Ebert, *Mater. Sci. Engg A.* 302,37 (2001).
- [3] H. Hu, *J. Mater. Sci.* 33, 1579–1589 (1998).
- [4] Sudarshan, M. K. Surappa, D. Ahn, and R. Raj, *Met. Mater. Trans A.*,39, 3291-3297 (2008).
- [5] Sudarshan, K. Terauds, A. R. Anil Chandra, and R. Raj, *Met. Mater. Trans. A.*, 45, 551-554 (2014).
- [6] P.G. Partridge, *J. Mett. Rev.*, 12, 169-194 (1967).
- [7] J. Li W. Xu, X. Wu, H. Ding, K. Xia, *Mater. Sci. Eng. A.*, 528, 5993-5998 (2011).
- [8] A. Chapuis, J. H. Driver, *Acta Mater.* 59,1986–1994 (2011).
- [9] S. Sandlobes, S. Zaeferrer, I. Schestakow, S. Yi, R. Gonzalez-Matinez, *Acta Mater.* 59, 429-439 (2011).
- [10] S. E. Ion, F. J. Humphreys, S.H. White, *Acta Mater.* 30,1909-1919 (1982).
- [11] B. Selvam, P.Marimuthu, R. Narayanasamy, V. S. Kumar, K.S. Tun and M. Gupta, *J. Mag. Alloys*, 3, 224-230, 2015.
- [12] X.J. Wang, X.S. Hu, K. Wu, K.K Deng, W.M. Gan, C.Y. Wang. M.Y. Zheng, *Mater. Sci. Eng A.*, 492, 481-485 (2008)
- [13] K.K. Deng, J.C. Li, F. J. Xu, K. B. Nie, and W. Liang, *Mater. Des.*, 67, 72–81 (2015).
- [14] Y.V.R.K. Prasad, K.P. Rao, and M. Gupta, *Comp. Sci. Tech.*, 69, 1070–1076 (2009)
- [15] L.H. Dai, L.F. Liu, Y.L. Bai, *Mater. Lett.* 58, 1773–1776 (2004).
- [16] Y. Yang, B.F. Wang, *Mater Lett.* 60, 2198-2202 (2006).
- [17] Jing Li, Haiyan Yang, Ping Yang, *Mater. Lett.* 134, 180-183 (2014).
- [18] N.M. Chelliah, P. Padaikathan, M.K. Surappa, *Mater. Sci, Engg: A*, 720, 49-59 (2018)
- [19] N.M. Chelliah, H. Singh, R. Raj and M.K. Surappa, *Mater. Sci. Engg A.*, 685, 429-438 (2017).
- [20] J.J. Fundenberger and B. Beausir, Universite de Lorraine, Metz, JTEX Software for Texture Analysis, <http://jtex-software.eu/>, 2015(accessed 20.11.2017)
- [21] S.L. Couling, J.F. Pashak, L. Sturkey, *Trans ASM*, 51, 94-107 (1959).
- [22] L.H. Dai, L.F. Liu and Y.L. Bai, *Mater. Lett.*, 58,1773–1776 (2004).
- [23] S.S. Zhou, K.K. Deng, J.C. Li, K.B. Nie, F.J. Xu, H.F. Zhou, and J.F. Fan, *Mater. Des.*, 64 177-184 (2014).

- [24] S. Suwas and R.K. Ray, Crystallographic Texture of Materials, (London, Springer, 2014) p.73-93.
- [25] G.W. Chang, C. Zhou, S.Y. Chen, *et al.*, Trans. Non-ferrous. Met. Soc. China, 20, 289 (2010).
- [26] Y.N. Wang and J.C. Huang, Mater. Chem. Phys., 81, 11-26 (2003).
- [27] R. Song, D. Ponge, D. Raabe, J. D. Speer, D.K. Matlock, Mater. Sci. Eng A., 441, 1-17 (2006).

Table. 1: Nominal composition of commercially pure magnesium for the present investigation

Matrix Material	Chemical Composition (wt%)						
Commercial Purity Mg ($\geq 99.9\%$)	Al	Mn	Si (Max)	Fe (Max)	Cu	Ni	Mg
	0.008	0.003	0.05	0.005	0.004	0.002	Balance

Fig 2: Microstructural evolution of deformed Mg-based composites after hot compression under different testing conditions

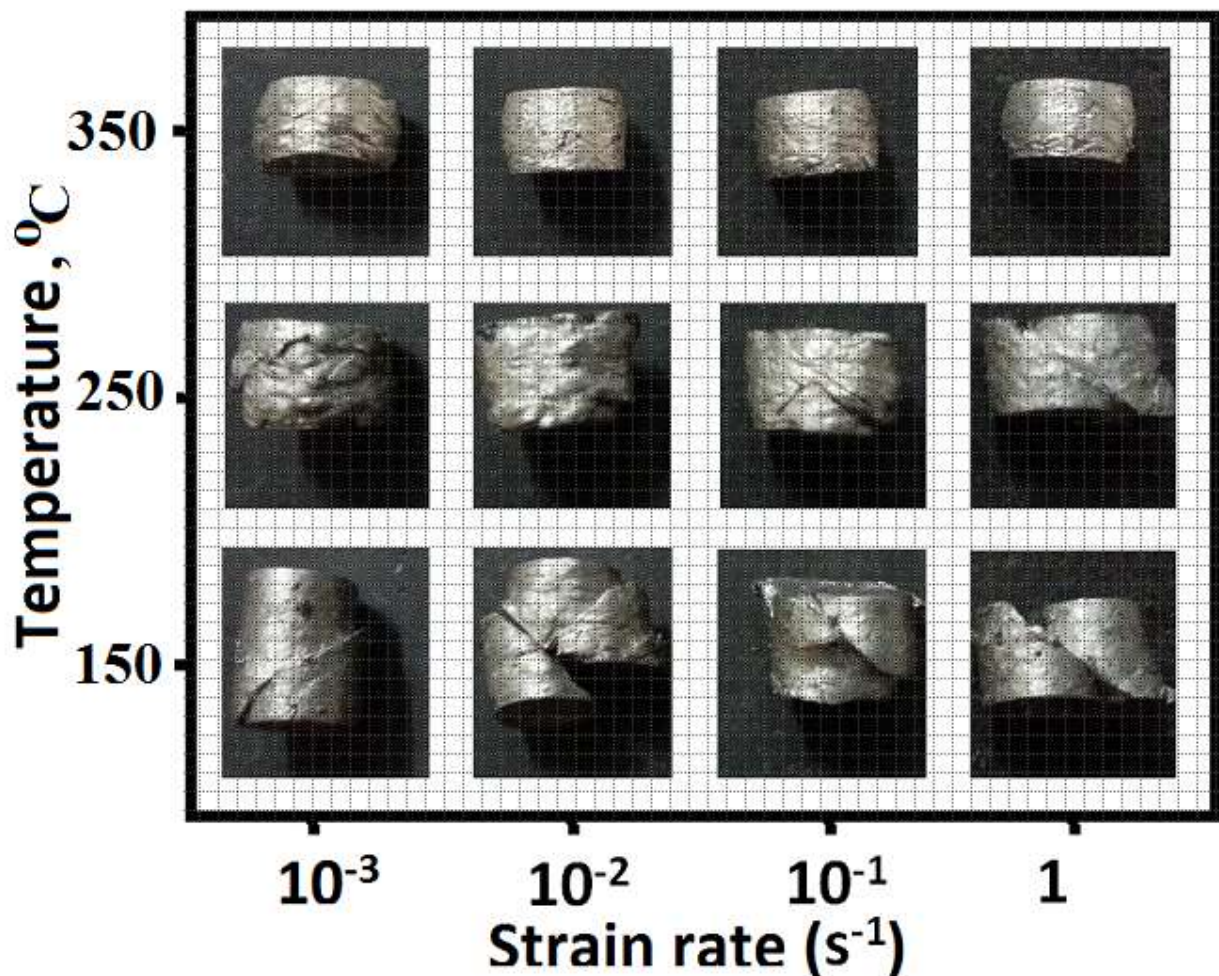


Fig 3: Macrographs of the deformed Mg-based composites after hot compression under different testing conditions

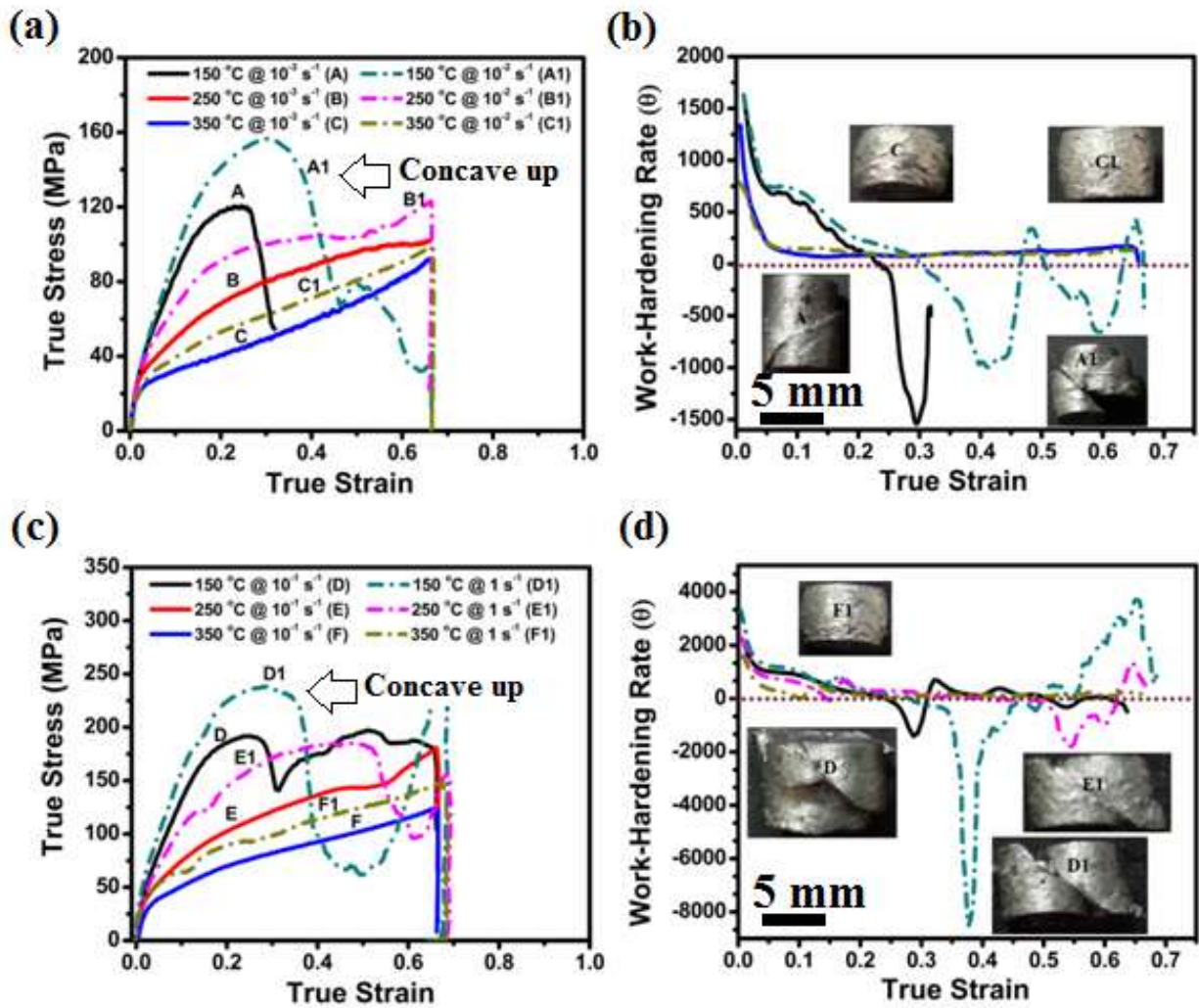


Fig 4 Hot compression behavior of the deformed Mg-based composites after hot compression under different testing conditions