Mechanical properties and cracking behaviour of hot dip galvanized ZnAlMg coatings.

Ahmadi, Masoud; Salgın, Bekir; Kooi, Bart; Pei, Yutao T.

Published in:
12th International Conference on Zinc & Zinc Alloy Coated Steel Sheet - GALVATECH 2021

MECHANICAL PROPERTIES AND CRACKING BEHAVIOR OF HOT-DIP GALVANIZED ZnAlMg COATINGS

Masoud Ahmadi[1], Bekir Salgın[2], Bart J. Kooi[3], Yutao Pei[1]

[1] Department of Advanced Production Engineering, Engineering and Technology Institute Groningen, Faculty of Science and Engineering, University of Groningen, Nijenborgh 4, 9747 AG Groningen, the Netherlands

[2] Tata Steel, Research & Development, P.O. Box 10000, 1970 CA IJmuiden, the Netherlands

[3] Nanostructured Materials and Interfaces, Zernike Institute for Advanced Materials, Faculty of Science and Engineering, University of Groningen, Nijenborgh 4, 9747 AG Groningen, the Netherlands

ABSTRACT

ZnAlMg coatings produced by hot-dip galvanization process have shown superior corrosion resistant, anti-galling and wear performances. Nevertheless, currently these coatings exhibit lower cracking resistance and ductility compared to conventional galvanized zinc (GI) coatings on steel sheets during forming processes. In this study, mechanical properties and cracking behavior of ZnAlMg galvanized steels have been investigated thoroughly. Microstructure, mechanical properties and key causes of cracking initiation and propagation have been scrutinized by utilizing scanning electron microscopy (SEM), orientation imaging microscopy, nanoindentation and in-situ SEM tensile/bending tests. Ultimately, effective plastic deformation-based factors are obtained to understand the cracking behavior and consequently link the microstructural features to cracking tendency of these coatings. The findings of this study are employed in designing new microstructure controlled ZnAlMg coatings with superb cracking resistance.

KEYWORDS

ZnAlMg coatings; Hot-dip galvanization; Cracking behavior; In-situ SEM tests, OIM

INTRODUCTION

Amongst zinc alloy coatings, hot-dip galvanized ZnAlMg coatings are well known for their extensive applications in construction and automotive industries in order to protect steel sheets. These coatings have offered outstanding corrosion and friction/wear performances in comparison with pure zinc (GI) coatings on steel substrates [1–5]. Nevertheless, cracking resistance and formability properties of these coatings are found to be lower compared to conventional zinc coatings.

Corrosion, composition, microstructure and hot-dip galvanization process parameters associated with these coatings have been studied in the literature [6–10]. There have been some efforts on the deformation and cracking of the conventional pure zinc coatings [11–14]. Yet there are limited studies in the literature concerning cracking behavior and formability of ZnAlMg coatings. Both Kollárová et al. [8] and De Bruycker et al. [15] carried out hardness evaluation experiments with varying contents of magnesium and aluminum. Kollárová et al. pointed out that the Knoop hardness was approximately doubled for the ZnAlMg specimen compared to the conventional zinc specimen. Much thicker cracks were observed in ZnAlMg microstructure after forming tests compared to pure zinc coatings. They also stated that better adhesion properties could be obtained by adding Al and Mg to the coating material. It was also reported that hardness significantly increases when aluminium is added to pure zinc and even more when the magnesium content is increased. They suggested a thermodynamic approach to provide better understanding about the solidification of the coating in hot-dip galvanization. Dutta et al. [16] investigated the morphology and microstructure of ZnAlMg hot-dip coatings. Hardness and scratch resistance of the coatings were increased by increasing magnesium content. Zunko et al. [17] focused on the effect of ZnAlMg coating thickness and steel substrate on cracking phenomenon. They claimed that thinner coatings deliver lower cracking density during deformation. An investigation on the cracking behavior of Zn2.5Al3Mg and Zn6Al3Mg coatings as the function of orientation was studied by Park et al. [16] using macro scale testing. They utilized electron backscatter diffraction (EBSD) analysis and found that crystallographic orientation of the primary zinc and eutectic phase affect the cracking tendency of the coating. They also indicated the presence of cracks before performing bending tests. They stated that the various coefficients of thermal expansion of the coating phases and the substrate generate tensile stresses along the planar direction of the coating that might influence the mechanical properties and cracking behavior of the coating. However, a clear understanding of cracking behavior as the function of different parameters yet remains unknown.

Comprehensive understanding of the cracking behavior of ZnAlMg coatings is still lacking. To achieve a thorough comprehension regarding the cracking behavior, it is vital to focus on in-situ micro-scale evaluations. Micro ductility and local orientation of the present phases can deliver helpful insights to explain the cracking initiation and propagation. In addition, the evolution of crack openings as well as the cracking pattern need to be quantified and studied by in-situ real time evaluations. This study focuses on these gaps and generates a groundwork for further investigations regarding cracking behavior of ZnAlMg coatings.

MATERIALS AND EXPERIMENTAL METHODES

For microstructural characterization, small samples were cut from the as-received Zn1.8Al1.8Mg coated high strength low alloy (HSLA) steel sheets with a total thickness of 0.6-0.62 mm produced by continuous hot-dip galvanization process (HDG). Samples were prepared by two polishing methods. First mechanical polishing using 1 μm diamond suspension and water-free lubricate on a Nap disc. Second, the mechanically polished sample was ion milled for 10-15 minutes with an ion polisher (JEOL IB-19520CCP) to attain a surface with good smoothness and quality for nanoindentation tests and electron backscattered diffraction (EBSD) analysis. The surface microstructure of the coating was studied by scanning electron microscopy (SEM, Philips XL30-FEG ESEM) and associated energy dispersive X-ray spectroscopy (EDS) mapping. EBSD analysis was performed in Philips XL30-FEG ESEM and postprocessing analysis of the data have been conducted using EDAX-TSL OIM software.

To assess the micro-scale mechanical properties of the present phases, load controlled nanoindentation experiments with the force of 3 mN are carried out using MTS Nano Indenter XP with a Berkovich tip at the continuous stiffness measurement mode. Appropriate spacing between the indents was applied in order to indent large number of the grains and phases.

To directly assess the real time cracking development, in-situ tensile tests were carried out employing Kammrath & Weiss (K&W) tensile module in EM. The dimensions of the tensile specimen gauge were as follows: 11.5 mm (length) × 3.5 mm (width) × 0.61 mm (thickness). The cracking behavior was also evaluated by in-situ bending (buckling) tests by means of the same stage on 20 mm × 7 mm × 0.61 mm specimens.

RESULTS AND DISCUSSIONS

One of the challenging aspects of characterization of ZnAlMg coatings is to obtain a good surface quality. Since typically the coating thickness lies around 10-15 µm, conventional bulk material preparation methods including mechanical grinding is not useful for the coatings. Yet, a helpful alternative is direct polishing of the sample. Fig. 1a illustrates the surface of the mechanically polished Zn1.8Al1.8Mg coating indicating three different microstructural components i.e. primary zinc (Zn), binary eutectic (Zn + $MgZn_2$), ternary eutectic (Zn + $MgZn_2$ + Al). As it can be observed, there exist some scratches over the grains due to mechanical polishing which makes the surface inappropriate for further EBSD and nanoindentation experiments. To overcome this, the sample is further ion polished and shown in Fig. 1b. As it can be noted, the microstructure is appeared much more smooth. The coating thickness is found about 15 µm by SEM cross sectional analysis given in Fig. 1c.

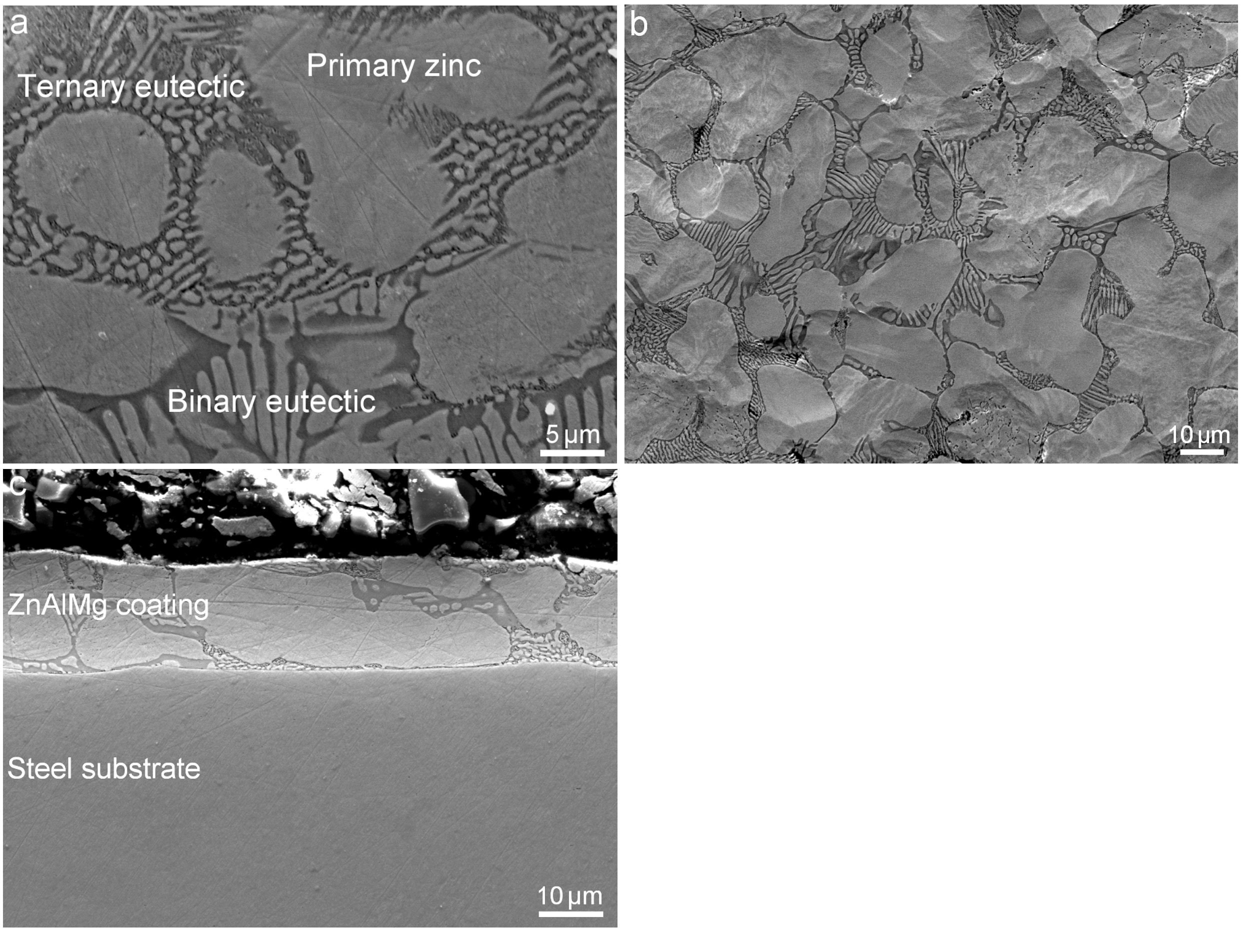


Fig. 1 (a) SEM micrograph of the surface of Zn1.8Al1.8Mg coating prepared by mechanical polishing; (b) SEM micrograph of the surface of the coating prepared by ion milling; (c) cross sectional SEM image of the coating.

In order to evaluate the mechanical properties associated with different microstructural component of the coating, nanoindentation tests were performed on the surface of the ZnAlMg coating. The indents along with EDS phase mapping are shown in Fig. 2a. Fig. 2b also depicts the corresponding SEM image of the selected area. As it can be noticed, all the existing phases are taken into account to obtain the corresponding local micromechanical properties. The size of the indents is found larger in the primary zinc grains compared to that in the eutectics.

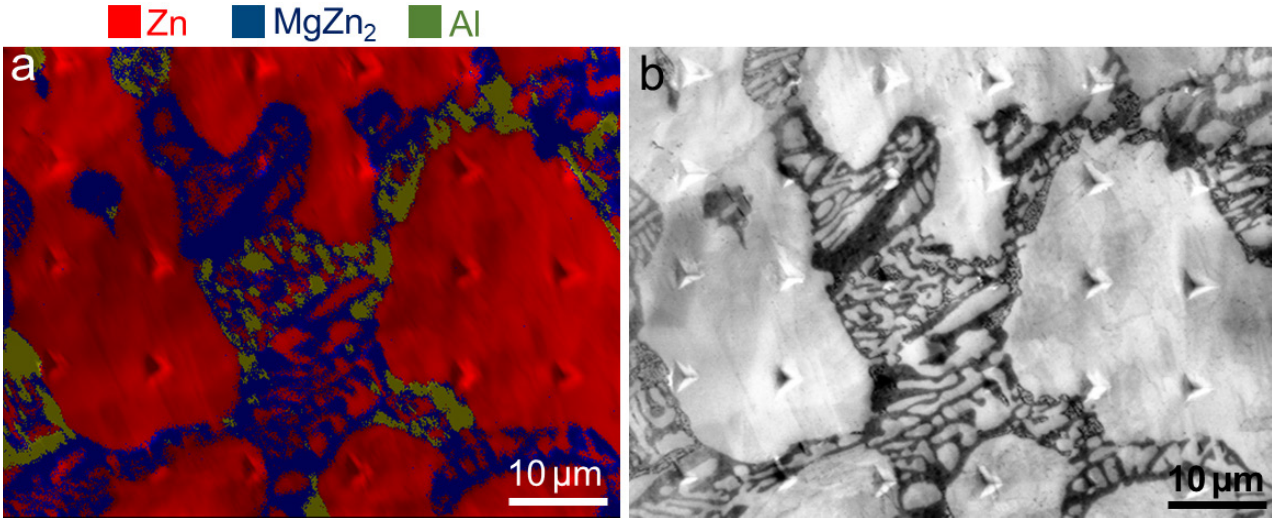


Fig. 2 (a) EDS phase mapping on the surface of nanoindented ZnAlMg coating; (b) SEM image of the selected area.

The results of the nanoindentation experiments are given in Table 1. As revealed, binary eutectic exhibits the highest hardness (H) but the lowest maximum penetration depth (p) and the lowest strain hardening exponent (n) among the microstructural components. This important finding indicates that, binary eutectic is the weakest component in terms of ductility and formability. Primary zinc phase delivers the best performance in terms of p and n values. One can perceive that, the contrast observed in the mechanical properties of the constituents is mostly attributed to the presence of hard but brittle $MgZn_2$ phase. The fraction and thickness of $MgZn_2$ intermetallic phase in binary eutectic are much larger compared to that of the ternary phase. Higher p and n values associated with the primary zinc and ternary eutectic indicate the higher tendency of these phases to endure plastic deformation and to offer higher ductility extent.

Table. 1 Average mechanical properties obtained by the nanoindentation experiments.

Phase	Youngs modulus (GPa)	Hardness (GPa)	Max. penetration depth, p (nm)	Strain hardening exponent, n
Primary zinc	108	1.5	328	0.34
Binary eutectic	92	2.7	242	0.08
Ternary eutectic	98	2.4	304	0.24

In addition to the local mechanical properties, crystallographic orientations of the microstructural components may play a significant role in ductility and consequently cracking tendency of the phases. For this purpose, EBSD analyses are performed on the ZnAlMg coating both before and after deformation. Fig. 3 shows the EBSD results of the coating before deformation. Fig. 3a represents the inverse pole figure (IPF) map along with the image quality (IQ) map of the investigated region of the coating. As it can be noted, the coating exhibits several random orientations revealed by different colors. However, the IPF texture representation of the coating given in Fig. 3c indicates that, most of the phases are oriented along [0001] and $[2\bar{1}\bar{1}0]$ orientation preferences. Many twinning can be observed in the IPF map. Moreover, phase fraction analysis by EBSD in Fig. 3b shows that, the majority of the coating microstructure is occupied by the primary zinc phases followed by magnesium zinc intermetallic ($MgZn_2$) and aluminium, which are in line with previous observation utilizing EDS mapping. EBSD analysis on the cracked regions will be furthered discussed in this study.

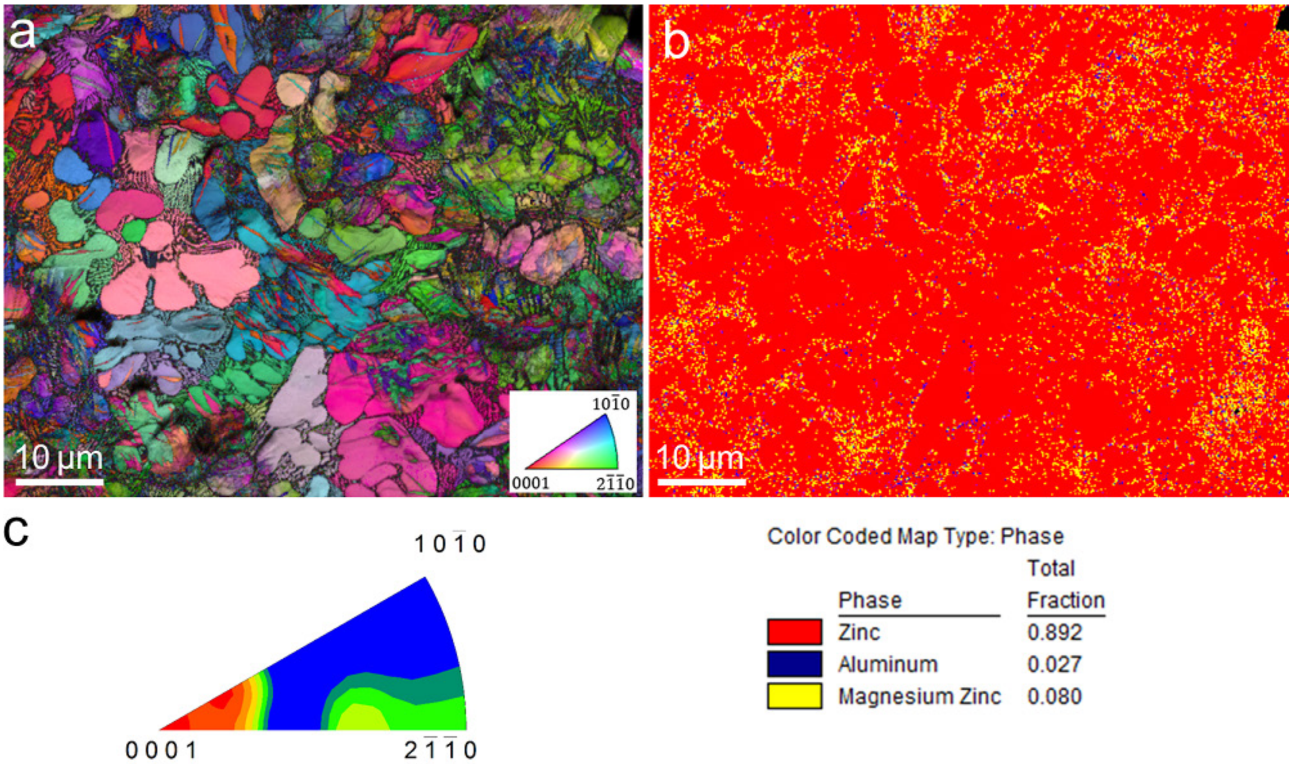
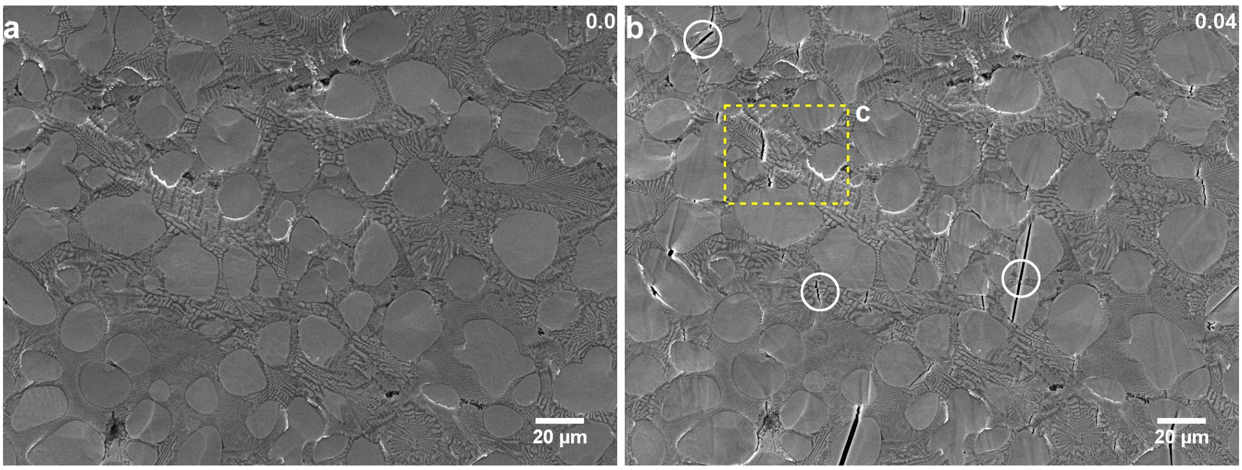


Fig. 3 EBSD results of the as-received Zn1.8Al1.8Mg coating: (a) inverse pole figure plus image quality map; (b) phase map; (c) IPF texture representation.

Cracking behavior of ZnAlMg coating is investigated by means of in-situ uniaxial tensile test. A random region (see Fig. 4) of the coating surface is selected and further tracked during the in-situ test. At global strain value around 0.04, the first micro cracks appear mostly in the binary eutectic namely the weakest microstructure component as already revealed by nanoindentation. This strain value lies in the Lüders band region of the steel substrate. Some of the cracks are further propagated through the primary zinc grains as a result of unfavourable crystallographic orientations with respect to the loading direction. The in-depth origin of micro-scale cracking have been examined and discussed by the authors [18]. Furthermore, average crack openings (width) are measured using image analysis and plotted versus the applied strain in Fig. 4f. As it can be observed, the crack opening is substantially increased as the deformation proceeds to higher strain values.



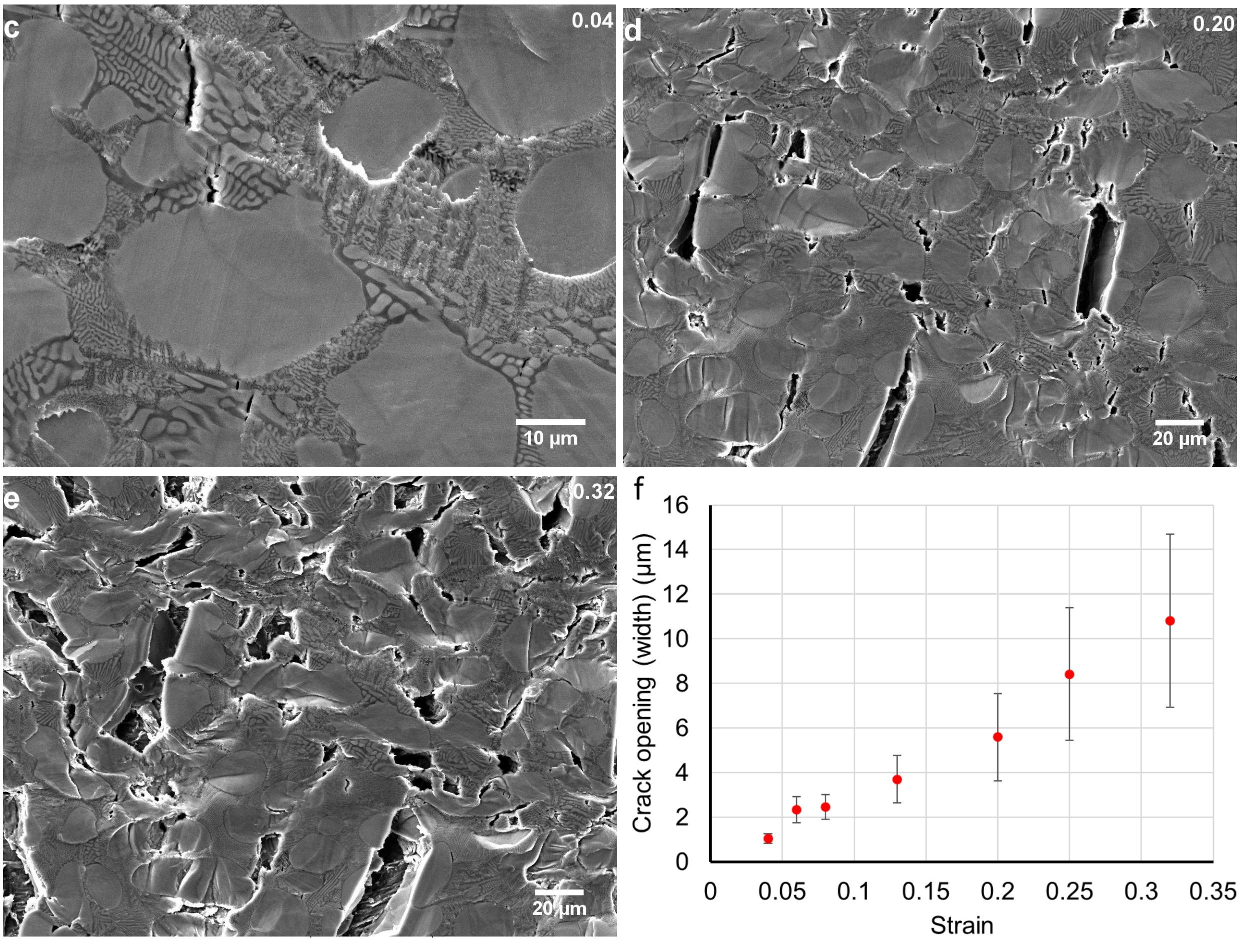


Fig. 4 In-situ tensile test results on Zn1.8Al1.8Mg coating: (a) selected region before deformation; (b) selected region at strain 0.04; (c) crack initiation in binary eutectic (d) selected region at strain 0.2; (e) selected region at the end of the test; (f) average crack opening versus applied global strain.

In order to evaluate the influence of crystallographic orientation on the cracking behavior of the microstructural constituents of ZnAlMg coating, EBSD analyses have been performed on the cracked and non-cracked areas. The results of the EBSD denote that phases aligned with [0001] parallel to the ND of the sample surfaces have managed to sustain cracking. On the other hand, some orientations are found as the detrimental and unfavourable orientation leading to cracking of the present phases. These unfavourable orientations normally exhibit low Schmid factor (m) and low strain hardening exponents (n) leading to cracking [18].

Bending mode deformation as an industrial relevant process is frequently used in the sheet metal forming. Therefore, the cracking behavior of the ZnAlMg coating is also investigated in bending tests. The results of the average crack opening (width) versus bending angle are plotted in Fig. 5. The corresponding top view SEM images of each stage of the bending are also illustrated. As it can be seen, by increasing the bending angle, more cracks are formed. It is significant to remark that, the formed cracks are further coalesced and formed larger cracks mostly aligned with the bending axis. At 0° bending angle, the sample is totally bent which corresponds to 0T bending mode used in industry. These large crack opening need to be reduced by implementing microstructural and processing parameter modifications in HDG process inspired by this study.

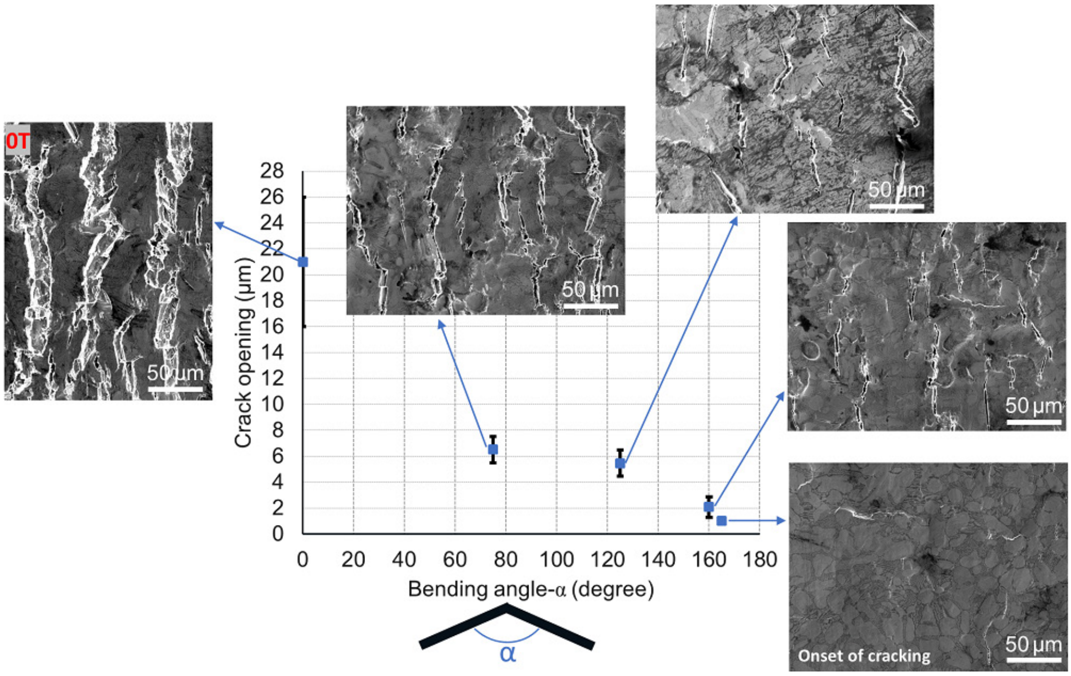


Fig. 5 Variation of the crack opening (width) versus bending angle of Zn1.8Al1.8Mg coating.

CONCLUSIONS

In this study, in-situ characterization and testing methods have been utilized to study the mechanical properties and cracking behavior of Zn1.8Al1.8Mg coating. Binary eutectic exhibits the lowest ductility and cracking resistance among the microstructure components of the coating. The orientations associated with the cracking tendency of the present phases have been investigated. Crack opening (width) has been correlated with strain values and bending angles. Larger cracks have formed in the bending deformation with a pattern mostly aligned with the bending axis. The findings of this study are applied for further comprehensive analysis on cracking phenomenon and designing a microstructure controlled ZnAlMg coating with excellent cracking resistance.

ACKNOWLEDGMEMT

This research was carried out under project number S22.3.15576 in the framework of the Partnership Program of the Materials innovation institute M2i (www.m2i.nl) and the Technology Foundation TTW (www.stw.nl), which is part of the Netherlands Organization for Scientific Research (www.nwo.nl).

REFERENCES

[1] T. Prosek, D. Persson, J. Stoulil, D. Thierry, Composition of corrosion products formed on Zn–Mg, Zn–Al and Zn–Al–Mg coatings in model atmospheric conditions, Corros. Sci. 86 (2014) 231–238.

[2] N.C. Hosking, M.A. Ström, P.H. Shipway, C.D. Rudd, Corrosion resistance of zinc–magnesium coated steel, Corros. Sci. 49 (2007) 3669–3695.

[3] D. Thierry, D. Persson, G. Luckeneder, K.-H. Stellnberger, Atmospheric corrosion of ZnAlMg coated steel during long term atmospheric weathering at different worldwide exposure sites, Corros. Sci. 148 (2019) 338–354.

[4] C. Yao, H. Lv, T. Zhu, W. Zheng, X. Yuan, W. Gao, Effect of Mg content on microstructure and corrosion behavior of hot dipped Zn–Al–Mg coatings, J. Alloys Compd. 670 (2016) 239–248.

[5] M. Van Schaik, C. Dane, B. Berkhout, MagiZinc-The New High Performance Coating for Steel in the BIW and Closures, SAE Technical Paper, 2016.

[6] Y. Chen, Y. Liu, H. Tu, C. Wu, X. Su, J. Wang, Effect of Ti on the growth of the Fe-Al layer in a hot dipped Zn-6Al-3Mg coating, Surf. Coatings Technol. 275 (2015) 90–97. https://doi.org/10.1016/j.surfcoat.2015.05.034.

[7] S. Farahany, L.H. Tat, E. Hamzah, H.R. Bakhsheshi-Rad, M.H. Cho, Microstructure development, phase reaction characteristics and properties of quaternary Zn-0.5Al-0.5Mg-xBi hot dipped coating alloy under slow and fast cooling rates, Surf. Coatings Technol. 315 (2017) 112–122. https://doi.org/10.1016/j.surfcoat.2017.01.074.

[8] M. Kollárová, L. Hrabčáková, J. Graban, M. Šohajová, Structural Properties of Zinc and ZnMgAl Coatings on Steel Sheets, Mater. Sci. Forum. 782 (2014) 623–626. https://doi.org/10.4028/www.scientific.net/MSF.782.623.

[9] Y. Xie, A. Du, X. Zhao, R. Ma, Y. Fan, X. Cao, Effect of Mg on Fe–Al interface structure of hot–dip galvanized Zn–Al–Mg alloy coatings, Surf. Coatings Technol. 337 (2018) 313–320. https://doi.org/10.1016/j.surfcoat.2018.01.038.

[10] H.R. Bakhsheshi-Rad, E. Hamzah, H.T. Low, M. Kasiri-Asgarani, S. Farahany, E. Akbari, M.H. Cho, Fabrication of biodegradable Zn-Al-Mg alloy: Mechanical properties, corrosion behavior, cytotoxicity and antibacterial activities, Mater. Sci. Eng. C. 73 (2017) 215–219. https://doi.org/10.1016/j.msec.2016.11.138.

[11] R. Parisot, S. Forest, A. Pineau, F. Grillon, X. Demonet, J.-M. Mataigne, Deformation and damage mechanisms of zinc coatings on hot-dip galvanized steel sheets: Part II. Damage modes, Metall. Mater. Trans. A. 35 (2004) 813–823.

[12] V. Di Cocco, F. Iacoviello, S. Natali, Damaging micromechanisms in hot-dip galvanizing Zn based coatings, Theor. Appl. Fract. Mech. 70 (2014) 91–98. https://doi.org/10.1016/j.tafmec.2014.05.003.

[13] G. Vincent, N. Bonasso, J.S. Lecomte, B. Colinet, B. Gay, C. Esling, The relationship between the fracture toughness and grain boundary characteristics in hot-dip galvanized zinc coatings, J. Mater. Sci. 41 (2006) 5966–5975. https://doi.org/10.1007/s10853-006-0274-6.

[14] G.M. Song, W.G. Sloof, Y.T. Pei, J.T.M. De Hosson, Interface fracture behavior of zinc coatings on steel: Experiments and finite element calculations, Surf. Coatings Technol. 201 (2006) 4311–4316. https://doi.org/10.1016/j.surfcoat.2006.08.046.

[15] E. De Bruycker, Z. Zermout, B.C. De Cooman, Zn-Al-Mg Coatings: Thermodynamic Analysis and Microstructure Related Properties, Mater. Sci. Forum. 539–543 (2007) 1276–1281. https://doi.org/10.4028/www.scientific.net/MSF.539-543.1276.

[16] M. Dutta, A.K. Halder, S.B. Singh, Morphology and properties of hot dip Zn-Mg and Zn-Mg-Al alloy coatings on steel sheet, Surf. Coatings Technol. 205 (2010) 2578–2584. https://doi.org/10.1016/j.surfcoat.2010.10.006.

[17] H. Zunko, A. Hackl, H. Antrekowitsch, R. Ebner, R. Brisberger, Investigation on Cracking Behaviour of new Zn-based Coatings on Sheet Steel for Hot Dip Galvanising, in: Proc. 0f EMC 2009. Eur. Metall. Conf. 2009; Innsbruck, Ger., 2009: pp. 1534–1536.

[18] M. Ahmadi, B. Salgın, B.J. Kooi, Y. Pei, Genesis and mechanism of microstructural scale deformation and cracking in ZnAlMg coatings, Mater. Des. 186 (2020) 108364.