ANALYSIS OF ZN-MG COATING IN DEPENDENCEON THE EXTENT OF DEFORMATION BY DIFFERENT TEMPERATURES

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Abstract

The Zn-Mg coatings are considered as the next generation of steel covering that should be used in automotive industry since they improve the corrosion resistance of the base material. The weakness of this coating lies in its higher hardness and lesser ability of plastic deformation in comparison to the purely zinc coatings. The Zn-Mg coatings break out and by higher deformation even flake off. This can influence the corrosion properties of the material. The experiment described in this article analyses the properties of the Zn-Mg coating in the dependence on the extent of deformation by different temperatures. The specimens were gradually deformed until their total rupture. Subsequently, the coating of deformed specimens was analysed by using electron microscope and the x-ray diffraction method. X-ray diffraction method is used for the evaluation of qualitative changes in the real structure of polycrystalline material. This method helps to find a suitable technology that do not cause cracking of the Zn-Mg coating.

Keywords: Coating, Deformation, X-ray Diffraction, Electron microscope

1. INTRODUCTION

The article deals with the analysis of a Zn-Mg coating of steel plates used mostly in automotive industry. In this experiment the changes of real structure and the surface disturbance of Zn-Mg coating in dependence on the degree of strain and temperature are analysed. [1,2] The experiment was carried out by using X-ray diffraction method, electron microscope analysis and static tensile test by using a temperature chamber. The main aim of the experiment was a comparison of results of two different methods (X-ray diffraction analysis and electron microscope analysis) used in this study. The specimens were standardized for the static tensile test by using the TIRATES 2300 machine. The specimens were 0.8 mm thick and covered with Zn-Mg coating. Individual specimens were loaded with a different tensile force that caused material strain. The experiment was performed at two temperatures, firstly, at the temperature of 20°C and the in the temperature of 250°C by using the temperature chamber.

2. EXPERIMENTAL CONFIGURATION OF THE MEASUREMENT DEVICE

In the first phase, the TIRATES 2300 machine with the temperature chamber was used to create requested strain of analysed specimens. Subsequently, Carl Zeiss ULTRA electron microscope was utilised to measure the thickness of the Zn-Mg coating and the degree of surface disturbance for various degrees of strain. To evaluate the changes in real structure of the material an *ISO DEBYEFLEX 3003* device, which uses Debye-Scherrer method, was used. [4]

2.1. THE ELECTRON MICROSCOPE CARL ZEISS ULTRA

The specimens were analysed by using the *Carl Zeiss ULTRA* electron microscope by acceleration voltages from 10 to 20 kV. The images were recorded by using detectors with filtering grid with high efficiency in - lens SE detector. Image processing can provide images in the quality up to 3072×2034 pixels. The machine uses the *Smart SEM* program control system. [3]

2.2. DEBYE-SCHERRER METHOD ANALYSIS

To analyse the changes in the real structure of material the backscattering arrangement of the Debye-Scherrer method was used. For this purpose, *ISO DEBYEFLEX 3003* device with non-filtered radiation from X-ray tube with chromium anode, cylindrical primary collimator 1 mm in diameter and image plate detector was employed. This method is non-destructive and, therefore, suitable for a qualitative evaluation parameters of real structure, i.e. for estimation of crystallite size, the extent of plastic deformation and also detecting the presence of texture. The advantage of the image plate detector lies in the simple processing of the image by using a scanner and the possibility of repeated use. The measurements were performed with 30 kV high voltage of the X-ray tube and electric current of 20 mA. The exposure time of the diffraction patterns from crystallographic planes *{112}* was 150 seconds. [5, 6]

3. THE SURFACE DISTURBANCE OF ZN-MG COATING IN DEPENDENCE ON THE DEGREE OF STRAIN AND TEMPERATURE

The specimens were first analysed by using the electron microscope that enabled monitoring the surface at a high magnification by retaining the sufficient depth of field.Subsequently, the specimens were analysed by using the X-ray diffraction method in order to find out the dependence of both methods by a certain deformation.

3.1. THE SURFACE DISTURBANCE OF Zn-Mg COATING AT THE TEMPERATURE OF 20 °C

The cold forming was applied to the steel plates covered with the Zn-Mg coating. [2] Therefore, the first experiment was performed at the temperature of 20 °C that is close to the ambient temperature. The specimens were deformed by the strain of φ=10 % and φ=45 %, which corresponds to the total damage of the material.

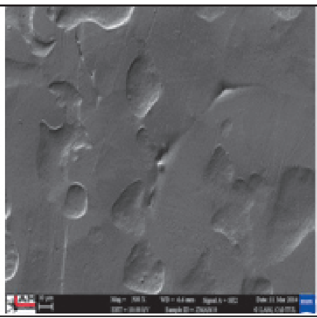
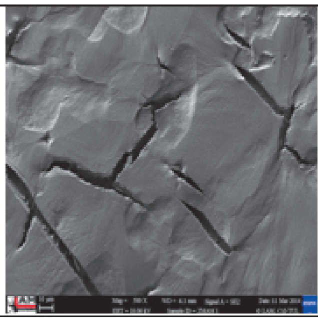
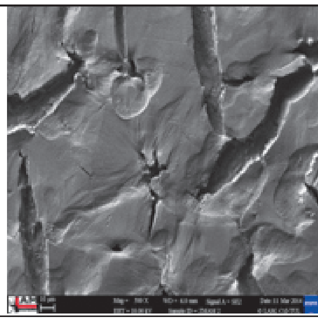
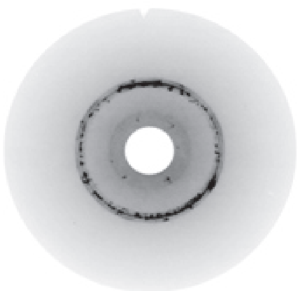
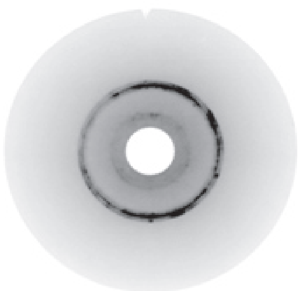
Electron microscope			
Strain φ [%]	**0%**	**10%**	**Maximum (45%)**
Debye-Scherrer method			
Strain φ [%]	**0%**	**10%**	**Maximum (45%)**

Fig. 1 The images of the surface layer in dependence on the degree of strain at the temperature of 20 °C

Fig. 1 shows a certain level of deformation by the strain of (φ=10 %). Even by this relatively low level of strain a disruption of the surface layer is caused. Because of its fragility, the Zn-Mg coating breaks out. By a higher strain the cranes are increasing. By the strain of (φ=10 %) the width of cracks is about 5 - 6 μm and by strain of (φ=45 %) the width of cracks exceeds 20 μm.

Based on the comparison of the results obtained by using the electron microscope analysis and the X-ray diffraction analysis certain correlation between the results of both techniques was found out. For the zero strain (φ=0 %) the diffraction line is discrete, which is characteristic for coarse-grained polycrystalline materials. In the case of the φ=10 % strain the diffraction line becomes “smoother” that is an evidence of plastic deformation. By this strain the surface was disturbed. With a rising strain the surface disturbance is growing and the back-reflection pattern is becoming continuous and shows the structure of a very fine-grained and plastically deformed material.

4. THE SURFACE DISTURBANCE OF ZN-MG COATING AT THE TEMPERATURE OF 250 °C

The second experiment was performed at the temperature of 250 °C. To reach this temperature the temperature chamber that is placed at the TIRATES 2300 machine was used. The specimens were placed to the temperature chamber to heat them sufficiently. Subsequently, the specimens were deformed by the strain of φ=7, 15 and 30 %.

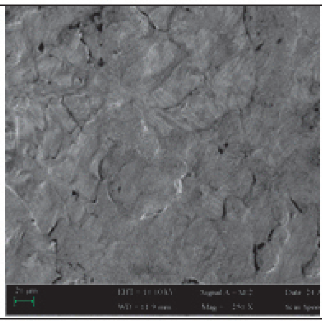
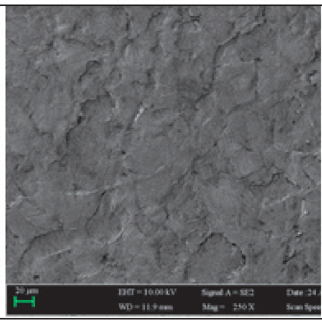
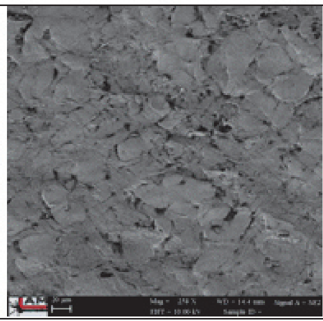
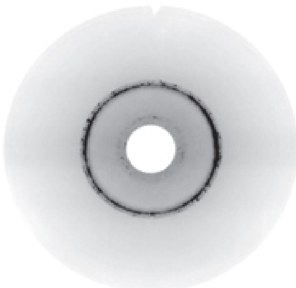
Electron microscope			
Strain φ [%]	**7%**	**15%**	**30%**
Debye-Scherer method			
Strain φ [%]	**7%**	**15%**	**30%**

Fig. 2 The images of the surface layer in dependence on the degree of strain at the temperature of 250 °C

The images obtained by using the electron microscope show that by the strain of φ=7 a 15 % no significant disruption of the surface layer was caused. The Zn-Mg coating is only deformed according to the plastic deformation of the steel sheet. By the strain of φ=30 % cavities are probably formed at the grain boundaries. The formation of cavities precedes the disruption of the surface layer. The higher temperature applied during the forming of steel plates covered with the Zn-Mg coating positively influence the quality of the surface layer.

The back-reflection patterns obtained by using the Debye-Scherrer method show that no significant changes in diffraction lines can be proved by individual strains. Based on a small change of individual diffraction lines it can be concluded that no significant disruption of the surface layer was caused.

5. CONCLUSION

For the Zn-Mg coating at the temperature of 20 °C it is typical that it is susceptible to the creation of cranes during the forming. **Fig. 1** shows a certain level of deformation by the strain of (φ=10 %). Because of its fragility, the Zn-Mg coating breaks out. By a higher strain the cranes are increasing. At the temperature of 250°C by the strain of φ=7 a 15 % no significant disruption of the surface layer was caused. (**Fig. 2**). The images obtained by using the electron microscope show no significant disruption of the surface layer: The Zn-Mg coating is only deformed according to the plastic deformation of the steel sheet. By the strain of φ=30% cavities are probably formed at the grain boundaries. By a higher strain a surface layer can be disrupted. The experiment has proved that the creation of cranes on the Zn-Mg coating can be eliminated by forming the material at higher temperatures. For a further research, the temperatures, by which the surface layer is not disrupted yet, will have to be determined.

The back-reflection patterns obtained by the Debye-Scherrer method show that at the temperature of 20 °C there is a change in the real structure of the Zn-Mg coating. Based on these results the Debye-Scherrer method could be applied to monitor the formation and subsequent development of the surface disturbance. The method is fast, simple, non-destructive and can be carried out on large specimens. The back-reflection patterns obtained by the Debye-Scherrer method show that at the temperature of 250 °C there is no significant change in the real structure of the surface layer. Therefore, it can be concluded that there is only a small disruption of the Zn-Mg coating.

The use of the steel plates covered with the Zn-Mg coating in practice cannot be unambiguously recommended, before additional tests and analyses are performed. These analyses should prove the corrosive stability of formed parts. In addition, other tests of individual forming technologies should be carried out because forming at the temperature of 250 °C is complicated and economically inefficient.

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REFERENCES

[1] DUTTA M., HALDER A. Morphology and properties of hot dip Zn-Mg and Zn-Mg-Al alloy coatings on steel sheet. Surface and rating Technology, Vol.205, Issue 7, December 2010, pp. 2578-2584.

[2] KOLL T., ULLRICH K, FADERL J., HAGLER J., SPALEK A. Properties and potential applications of ZnMg-alloy-coatings on steel sheet by PVD. In: Proceedings of Galvatech '04, Association for Iron & Steel Technology, Chicago, 2004.

[3] Ultra plus. Imaging & Microscopy [online]. 2009 [cit. 2014-04-26]. Available from: http://www.imaging-git.com/products/ultra-plus-ultra-high-resolution-imaging-non-conductive-samples

[4] KRAUS I., GANEV N. Residual Stress and Stress Gradients, In: Industrial Applications of X-Ray Diffraction. New York. 2000, pp. 793-811.

[5] STANCEKOVA D., SEMCER J., DEBRAS M., KURNAVA T. Methods of Measuring of Residual Stresses and Evaluation of Residual State of Functional Surfaces by X-Ray Diffractometric Methods, Manufacturing Technology, Vol. 13, No. 4, 2013, pp. 547-552.

[6] KRAUS I., GANEV N. Technické aplikace difrakční analýzy. ČVUT. Praha 2004.