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In-Situ Characterization of the Mechancial Behavior of Hot-Dip Zn-5al Coating: An Overview of Deformation and Damage Mechanisms

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Zinc-based coatings are used in diverse applications mainly for their high resistance to corrosion and other properties such as good longevity. Previous research has focused mainly on microstructure and corrosion resistance of these coatings. However, mechanical properties are seldom mentioned in literature. Only a few studies have investigated mechanical behavior of zinc-based coatings, especially those exhibiting complex microstructures after addition of elements. This work focused on a Zn-5Al coating deposited on steel by continuous hot-dip galvanizing, giving a detailed overview of its microstructure and its crystallographic texture. Interesting deformation and damage mechanisms were revealed through in-situ tensile experiments coupled with digital image correlation measurements. Crack distribution and chronology of deformation and damage events were determined and well understood. In this experimental work, diverse techniques ranging from microstructure analysis with SEM and EBSD to the study of the mechanical behavior using micro-mechanical tensile testing, in-situ SEM observations and DIC measurements as well as advanced techniques such as focused ion beam for more in-depth studies were considered. The purpose of this work is to give a better understanding of deformation and damage mechanisms of zinc-based coatings.

Keywords: *Hot-dip galvanizing, Microstructure, Mechanical behavior, Deformation, Damage*

1. Introduction

Zn-5Al coating is characterized by a particular microstructure with pro-eutectic zinc dendrites and eutectic phases [1]. This microstructural heterogeneity gives to the coating particular mechanical properties [1,2,5,6]. Several works highlighted the role of addition of aluminum to the zinc bath, especially regarding the formation of the inhibition layer that prevents the formation of the brittle Fe-Zn phases [3]. However, no particular interest has been given to the understanding of the deformation and damage modes of this coating.

This work aims to define the chronology of the deformation and damage events of this coating under uniaxial tensile loading. First, an overview of the different experimental techniques is given. Then, an in-depth analysis of the coating's microstructure and its crystallographic texture is performed. The last part is a discussion of the different observations and results and an interpretation of the deformation and damage events.

The experimental work combines multiple advanced techniques ranging from microstructural characterization to mechanical testing. The originality of this work is not only due to the technical findings, but also to the way the different characterizations techniques are brought together to understand the mechanical behavior of the coating.

2. Experimental Procedure

The initial state of the coating is studied using scanning electron microscopy (SEM). This gives insights on the different phases and the microstructure of the coating. The crystallographic texture is analyzed by means of

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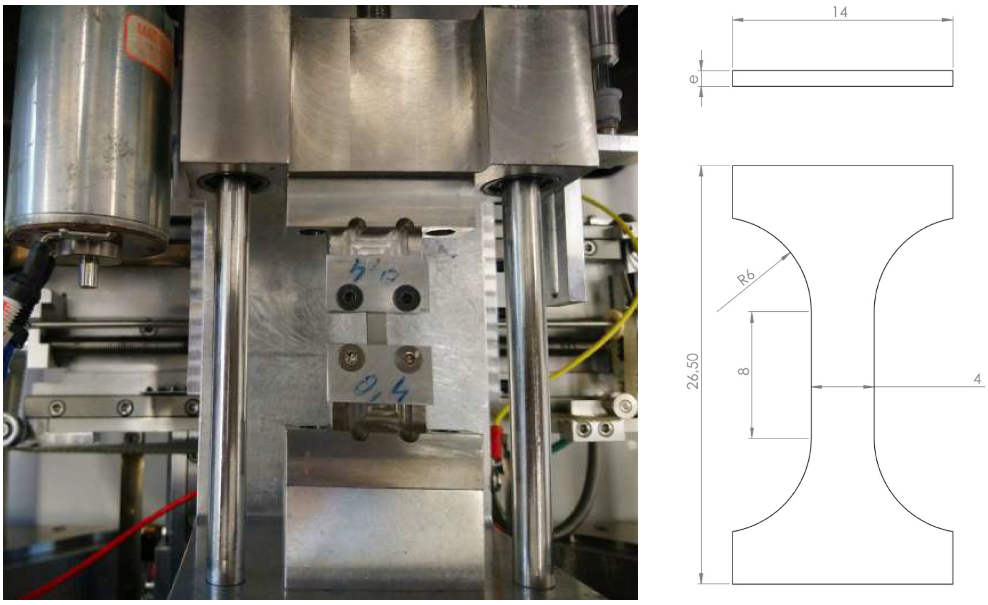

Fig. 1. In-situ SEM tensile test configuration and specimen geometry, dimensions in mm

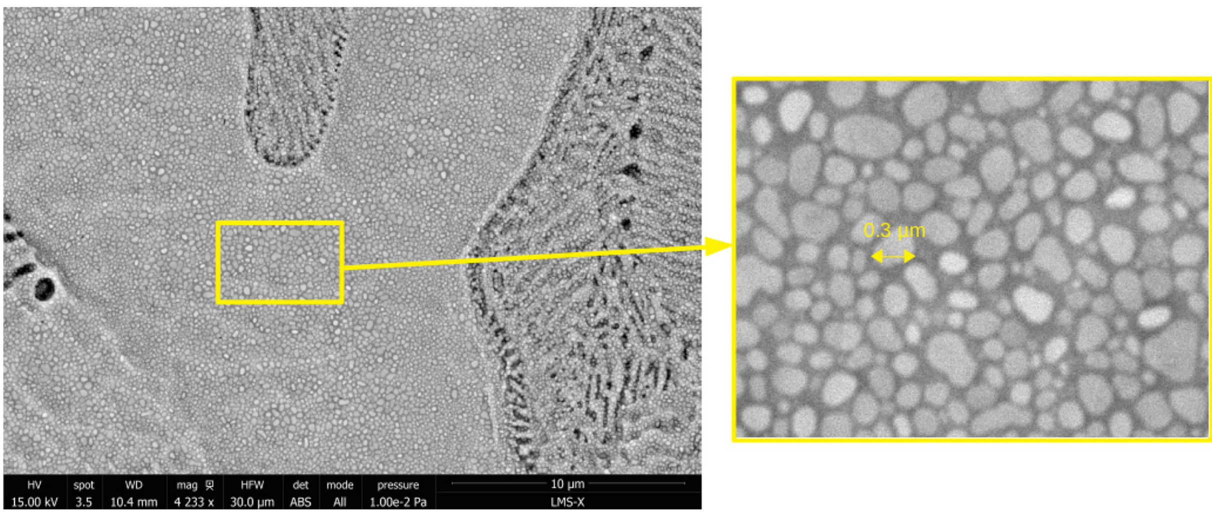

Fig. 2. Tin speckle applied on the surface of the coating for DIC measurements

electron backscatter diffraction (EBSD), giving information about the orientation of grains, which is an important element to consider for understanding the mechanical behavior of the coating, especially that the initial texture of the material contributes to the definition of its micro-mechanical behavior.

In-situ tensile test is performed to assess the chronology of deformation and damage events of the coating. An overview of the test setup and the geometry of the specimen is shown in Fig. 1. Elongation is applied and SEM images are captured at different straining levels. In addition, it is important to capture strain fields during the tests. Therefore, a speckle is applied on the surface of the coating to enhance the contrast allowing to perform digital image correlation (DIC).

An example of the obtained speckle (Sn speckle, 20 nm layer) is shown in Fig. 2. This technique allows to create nanoparticles with the size of 300 nm, leading to an enhanced contrast and a much robust and easier correlation of images [1,4,7,8]. Before the application of this layer, the sample is prepared using ion polishing technique. The latter is essential to achieve a well-prepared sample and to get high indexation rates when performing EBSD.

During the test, 19 steps are considered: an initial state corresponding to 0% of elongation, 4 supplementary steps up to 1% of elongation and then images are captured for each additional 1% of elongation until reaching a final level of 15%. Post-processing of the obtained images is performed afterwards to obtain strain fields that help in identifying the type and the order of the deformation and damage events.

Focused ion beam (FIB) is used after the test to carry out more advanced characterizations either to understand the microstructure of the coating or to investigate the path of cracks along the thickness of the coatings.

The steel substrate considered for this work has a thickness of 0.55 mm and the coating thickness is 7 µm.

3. Microstructre and Crystallographic Texture

3.1 Microstructure

The microstructure of the Zn-5Al coating is shown in Fig. 3. SEM image (BSE) showing the microstructure of Zn-5Al coating. One can see that there are 2 phases: pro-eutectic zinc dendrites and a binary eutectic phase, which can be either lamellar or globular. During solidification, zinc dendrites nucleate first, and the remaining liquid undergoes the eutectic transformation. This results in having a zinc matrix with a globular or a lamellar eutectic (Al-rich phase). The latter undergoes afterwards the eutectoid transformation where it is further decomposed into zinc and aluminum. This process happens at different locations and creates multiple solidification cells (flake structure).

The difference of morphologies for the eutectic phase (lamellar and globular) remains challenging to be fully understood. However, a possible explanation for this is the presence of pro-eutectic zinc dendrites which can act as a source of disturbance for the liquid during solidification, causing this change in morphology. In other words, the zinc dendrites can alter the thermodynamics of the solid-liquid interface and the orientation and growth of eutectic phases.

Fig. 4 is a sketch summarizing the solidification process of Zn-5Al coating and how the final microstructure is obtained.

3D reconstruction of the microstructure of this coating is achieved and is shown in Fig. 5. This observation shows that the globular and lamellar eutectic phases can

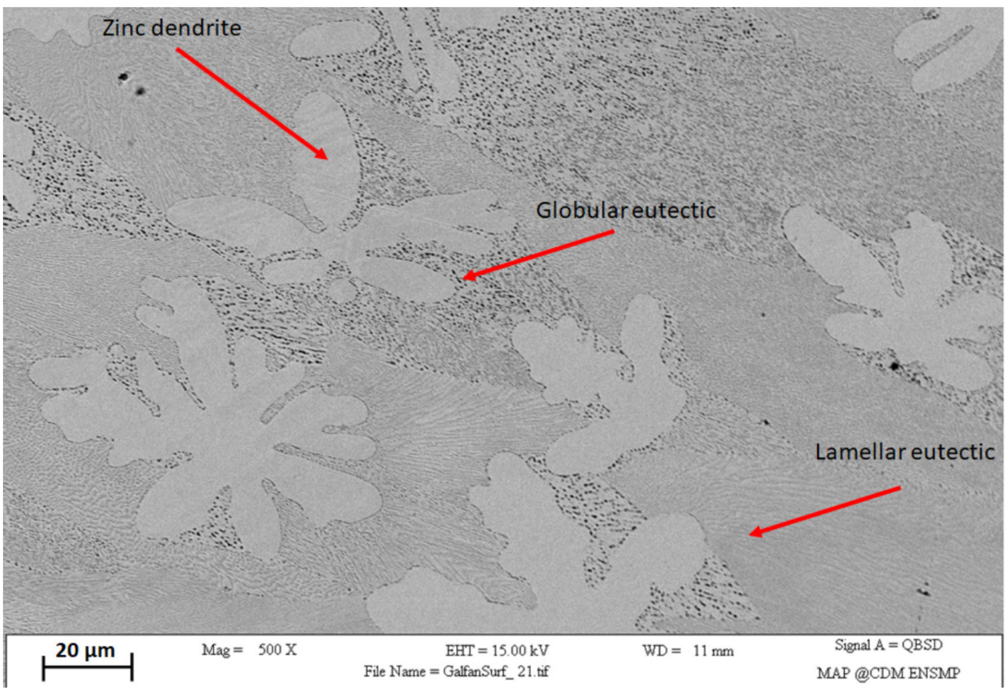

Fig. 3. SEM image (BSE) showing the microstructure of Zn-5Al coating

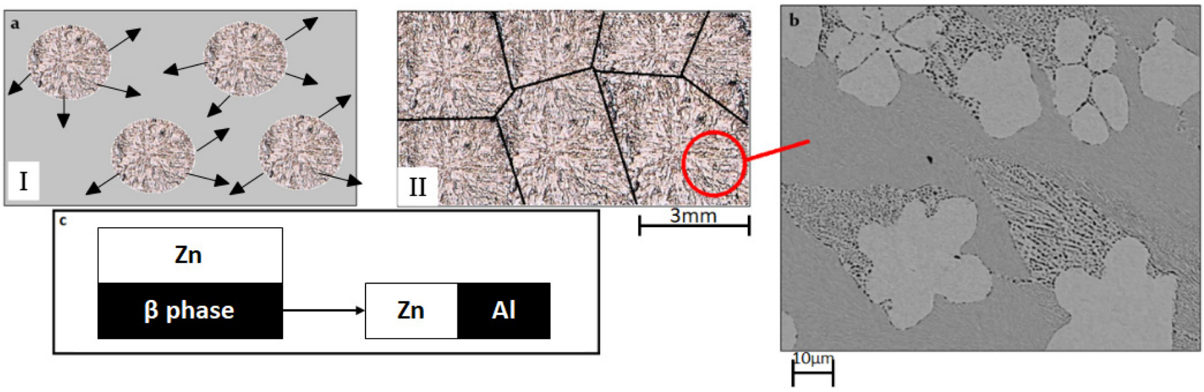

Fig. 4. Summary of the microstructure of Zn-5Al coating: (a-I) Sketch of radial growth process of flake structures and (a-II) formation of solidification cells, (b) Dendritic and eutectic structures, (c) Eutectoid transformation

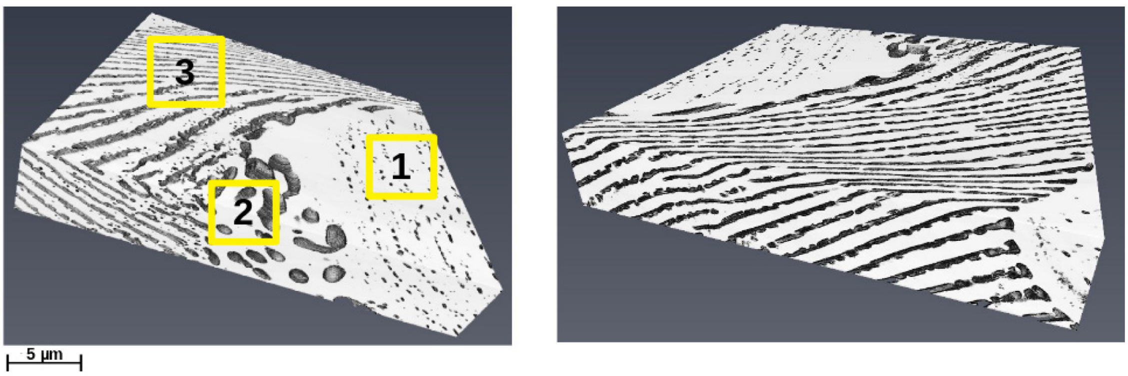

Fig. 5. 3D reconstruction of Zn-5Al coating: (1) Zinc dendrite, (2) Globular eutectic and (3) Lamellar eutectic

be the same phase but oriented differently. In fact, if the lamellar eutectic is considered as a set of parallel rods, and an orthogonal cross-section is performed, these rods could appear as circles and look like the globular eutectic.

3.2 Crystallographic texture

The crystallographic texture of the coating is shown both at global and local scales in Fig. 6 and Fig. 7 respectively. The IPF-Z maps show that this coating is characterized by a strong basal texture but can have some orientation heterogeneities at a local scale. These local heterogeneities can influence drastically the machinal performance of the coating since they will be associated to different deformation and damage mechanisms.

The average grain size of the coating is 40 μm. One interesting finding is the epitaxy relationship between the zinc dendrites and the eutectic present in their vicinity. It seems that zinc dendrites impact the orientation of the surrounding eutectic and therefore a grain is formed by a mixture of pro-eutectic zinc and eutectic.

3.3 Solidification model

In the following, an interpretation of the microstructure combined with the EBSD analyses is performed to suggest a possible solidification model for Zn-5Al coating.

For the formulation of this model, two mechanisms are considered. The first one concerns zinc growth, whether it is a primary dendrite or within a eutectic phase: this growth occurs more rapidly in the dense directions of the hexagonal lattice, which are the 6 directions <001> of the hexagonal plane (001). The second one concerns the solidification front of the eutectic phase: it is always perpendicular to the direction of growth of the beta-lamellae. The beta phase can be separated in the form of planes or rods perpendicular to

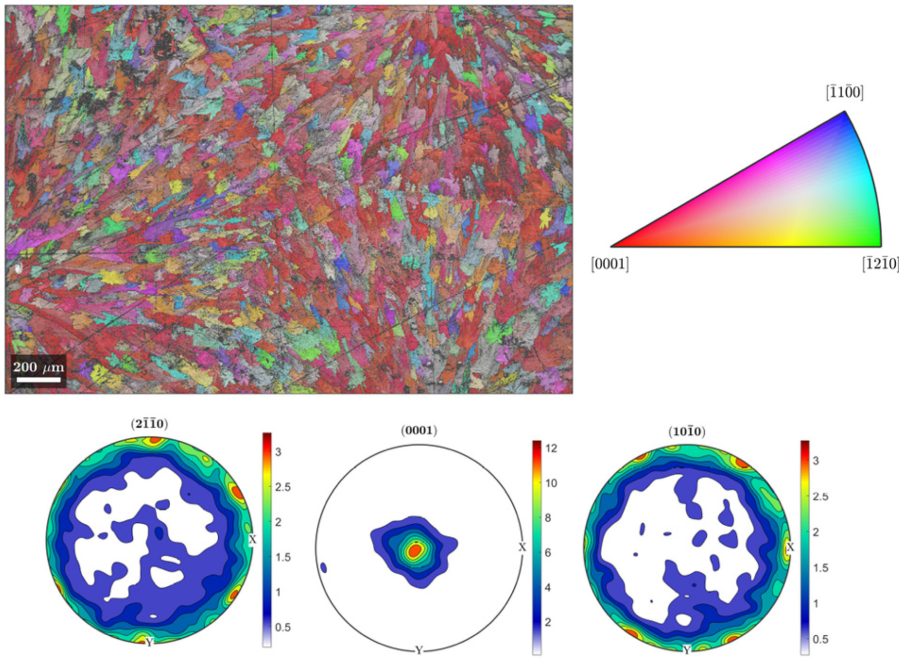

Fig. 6. Crystallographic texture analysis of the surface of Zn-5Al coating – global view, Z=ND

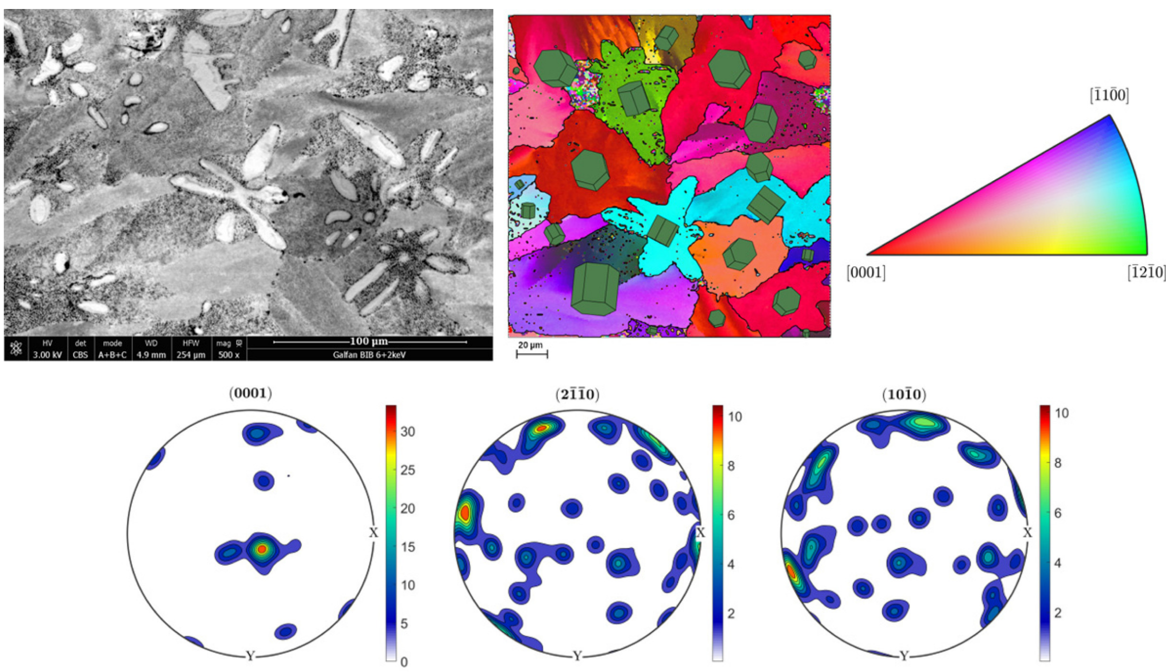

Fig. 7. Crystallographic texture analysis of the surface of Zn-5Al coating – local view, Z=ND

the solidification front.

Relying on the EBSD analyses, it is confirmed that the coating has a dominant basal texture. Therefore, zinc grains with their c-axis perpendicular to the plane of the sheet will grow faster. Since an epitaxial relationship exists between the primary zinc dendrites and the eutectic phase, this latter propagates more if it is associated to a basal zinc dendrite. The solidification front propagates while remaining perpendicular to the plane of the sheet and the free surface. The size of the grains will then be limited by the encounter of another solidification front. The size of these grains is therefore highly related to the number of nuclei and to the cooling rate when crossing the liquidus.

During solidification, all primary zinc nuclei can give rise to a eutectic that is epitaxially related to it. If this eutectic does not have a basal orientation, its extension will be limited by the thickness of the coating. In this case, it is expected that the beta phase (rods-like structure) appears as globules: this eutectic is observed via a plane perpendicular to the direction of growth of the rods, explaining the observations made for the three-dimensional geometry of the coating.

In the other case, where the eutectic grows with a basal texture, the beta and zinc phases in the eutectic are separated into planes rather than rods, possibly because this solidification is rapid, and the system then observes that it is much easier to make planes than rods to grow fast. An effect of the interface energies between beta and zinc phases in the eutectic can be an explanation for this phenomenon. In fact, if solidification is fast, the aluminum phase can afford to have greater surface energy in lamellae than in rods structures.

This short formulation gives a possible explanation for the appearance of different eutectic structures, for instance globular and lamellar, and establishes a link between the observed microstructure, the solidification and the crystallographic texture of the coating.

4. Mechanical Behaviour of the Coating

During the in-situ tensile test, SEM images are acquired for two different horizontal field widths. The first one is 800 μm and the second one is 150 μm. These two different levels stand for mesoscopic and microscopic observations, respectively. The image resolution is kept

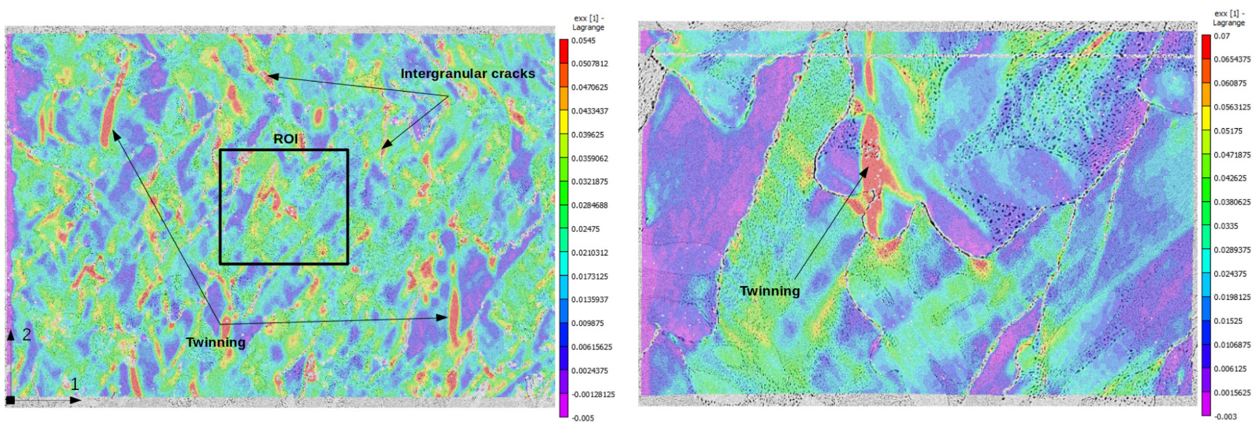

Fig. 8. Strain field measurements at 1% of macroscopic strain, loading direction is horizontal

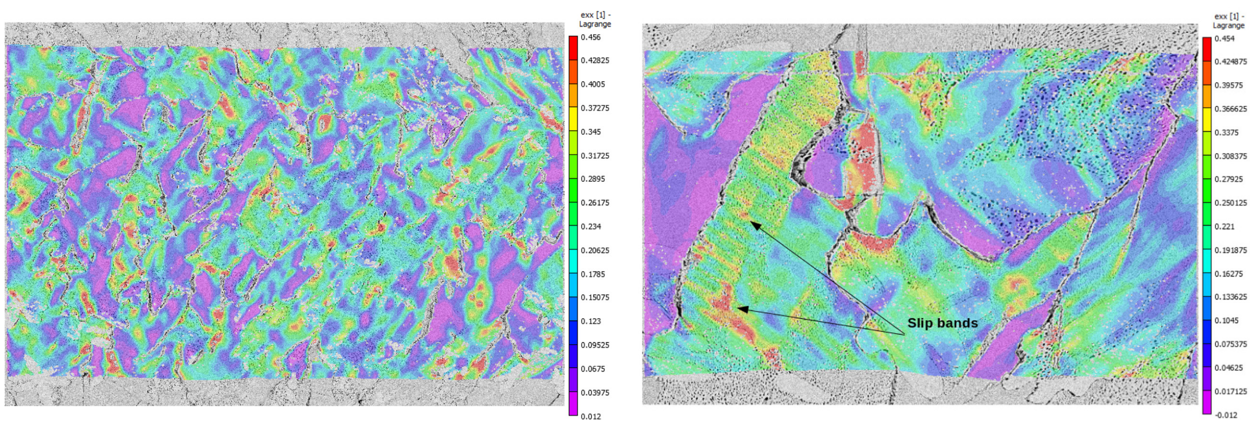

Fig. 9. Strain field measurements at 15% of macroscopic strain, loading direction is horizontal

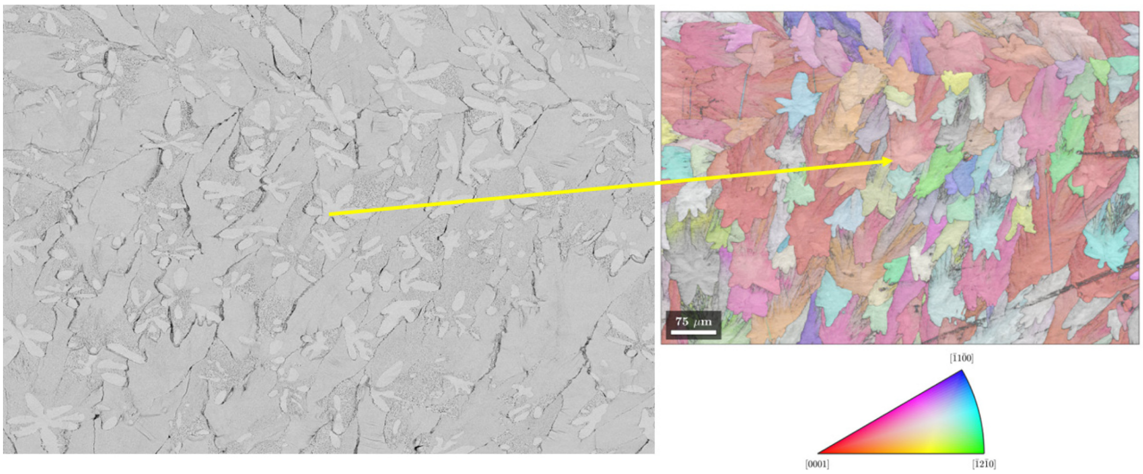

Fig. 10. Intergranular cracking mechanism for Zn-5Al coating

the same, allowing to detect more local events for the images with the smallest field width.

Fig. 8 and Fig. 9 show the strain field measurements obtained after 1% and 15% of macroscopic strain, respectively, for the two field widths. The observed strain localization is originating from growth of twinning bands and intragranular decohesion. The latter is found to be the dominant damage mechanism of the coating. It initiates at rather low strain (1% of macroscopic imposed strain, local strain is 5% based on the measurements at HFW = 800 μm). Then, propagation and opening of cracks is observed up to the maximum applied strain (15% of macroscopic strain, 45% of local strain). By the end of the tensile test, almost all grain boundaries have cracks. Growth of twinning bands is detected since the first stages of straining (1% of macroscopic strain, local strain is 7%). These twinning bands are either present in the as deposited state of the coating or form during straining to accommodate the deformation. These twinning bands become detrimental for the coating. In fact, since twins are oriented in a way that promotes cleavage cracking, they act as a trigger for fracture. However, these cracks seem to grow at twin boundaries and not inside the twins. Another observed mechanism is activity of slip systems: slip bands are observed. This gliding is another mechanism allowing to accommodate the deformation.

The main deformation and damage mechanisms of Zn-5Al coating are identified: Intergranular cracking is the main damage mechanism. This is further detailed in

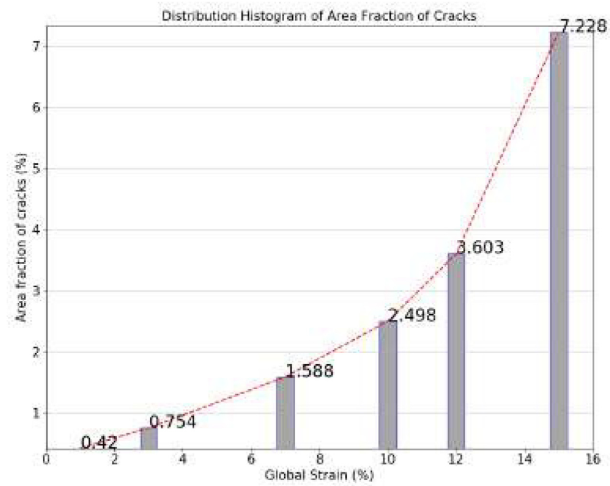

Fig. 11. Evolution of the area fraction of cracks with respect to the applied strain level

Fig. 10 where SEM image of the sample after 15% of macroscopic strain is compared to the EBSD analysis of the same area prior to straining. Cracks form mainly at grain boundaries. The evolution of the area fraction of cracks is illustrated in Fig. 11. This fraction is defined as the ratio between the area of cracks to the total area of the analyzed surface. One can see that with the increase in the strain level, the area fraction of cracks becomes more important. This is caused either by propagation of existing cracks or the formation of new cracks.

As for the deformation mechanisms, both twinning and gliding are observed. Fig. 12 displays an area having a twin that is present at the deposited state. With

Fig. 12. SEM image of Zn-5Al coating after 15% of macroscopic strain showing a crack forming at twin boundaries

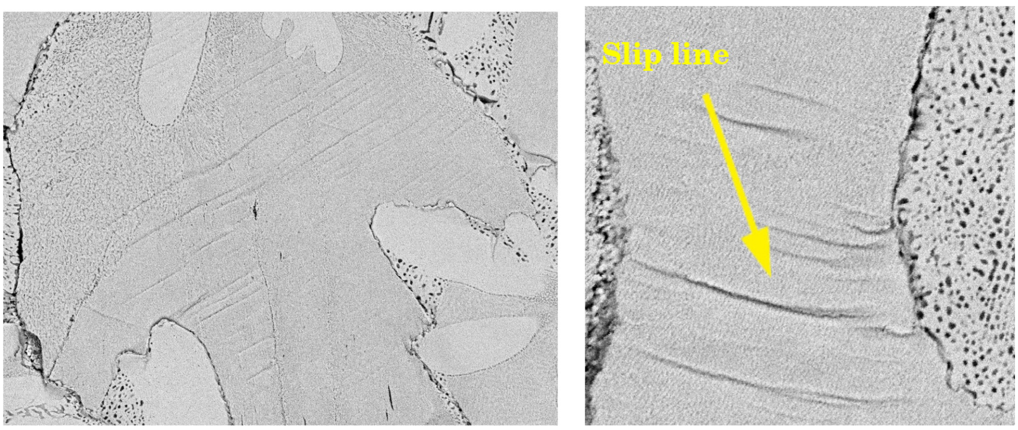

Fig. 13. Slip bands observed after straining

increasing the strain level, the twin band grows. Then crack initiation is observed. SEM images observations suggest that these cracks are located at twin boundaries. Slip bands are clearly observed in Fig. 13. These bands act as another way for strain accommodation. Grains that cannot twin will deform plastically by forming these slipping bands.

The central area of the zone of interest considered for the tensile test is analyzed further using FIB. This area represents almost all the deformation and damage mechanisms observed for the coating: intergranular fracture, twinning, and its associated fracture. Slices are performed and are shown in Fig. 14. One can see that even for such a high and severe strain level, the cracks do not reach the steel substrate and remain confined near the free surface of the coating. Good adhesion is also observed. This observation shows that intergranular cracks can differ in terms of sizes and openings. The twin inside the dendrite causes change in local roughness of the coating and the associated crack appears to be forming at the boundaries of this twin.

5. Conlusion

The work highlighted the microstructure and the crystallographic texture of the coating and presented a possible solidification model of Zn-5Al coating. It also described the protocol followed to build a better understanding of the mechanical behavior of Zn-5Al coating.

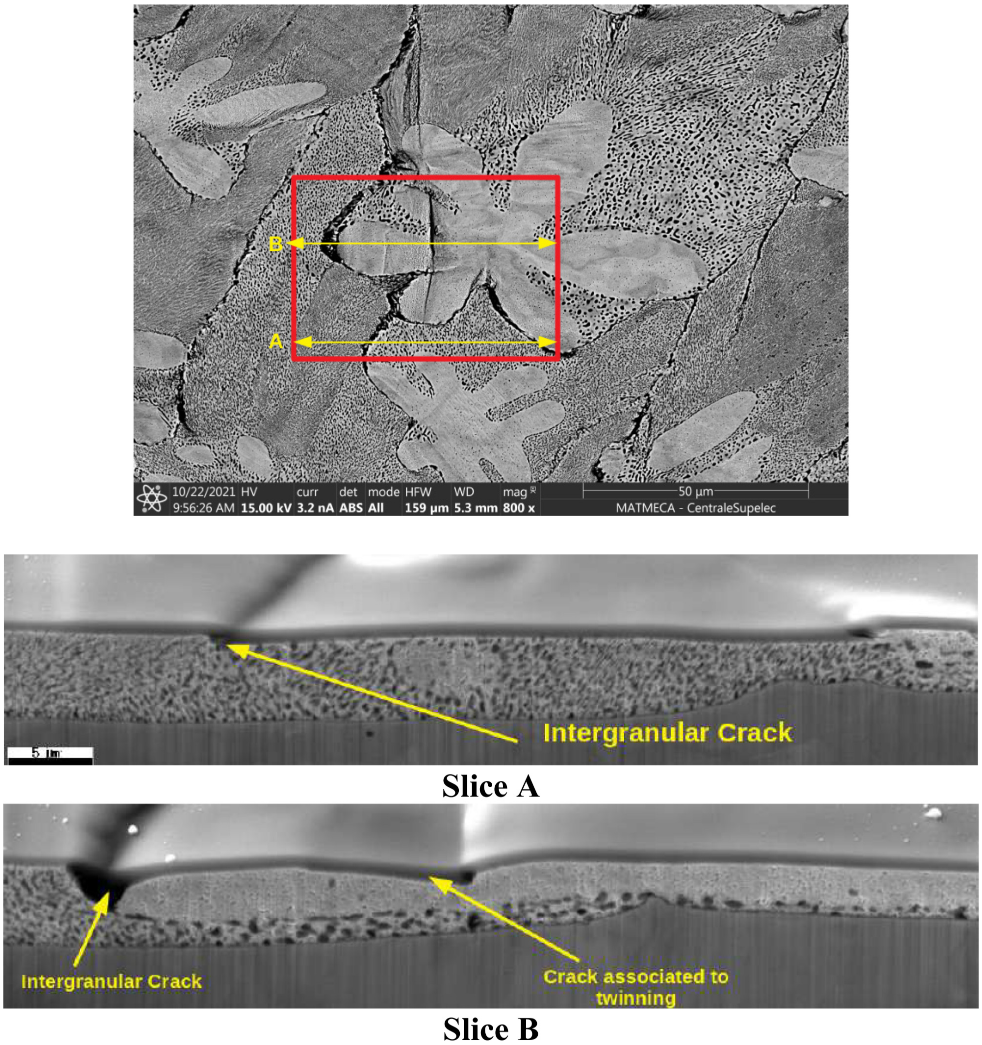

Fig. 14. FIB analysis on Zn-5Al coating after 15% of macroscopic strain

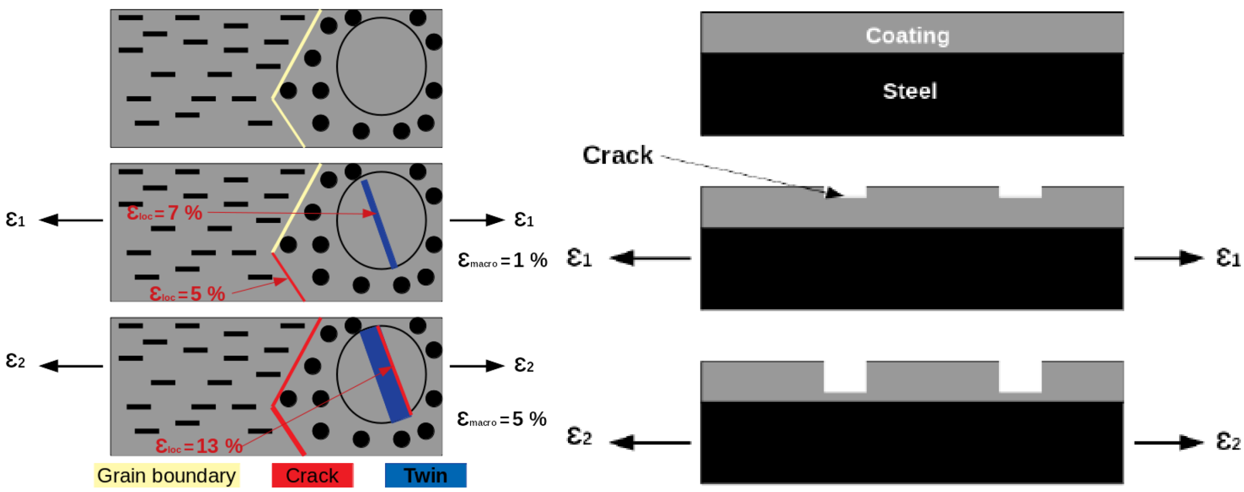

Fig. 15. Sketch of the deformation/damage mechanisms of Zn-5Al coating, $\varepsilon_1 < \varepsilon_2$, Local strain thresholds are determined using the DIC results for a given macroscopic strain level

The deformation and damage mechanisms of the coating are now better understood. This is achieved through advanced in-situ tensile testing. A summary of the observed events is illustrated in Fig. 15: At first stages of straining, grain boundary decohesion is observed. Twinning also takes place. With the increasing strain, cracks become larger and are widened. They propagate slowly towards the steel substrate. Twins undergo cracking at their boundaries and cleavage cracks are rarely observed.

Cross-sectional observations reveal good adhesion of the coating and that cracks remain close to the free surface of the coating.

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