Evolution of deformation heterogeneity at multiple length scales in a strongly textured zinc layer on galvanized steel

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Abstract. The evolution of heterogeneity of plastic deformation in a zinc layer has been probed at multiple length scales using a battery of characterization tools like X-ray diffraction, electron back scatter diffraction (EBSD) and digital image correlation. The experimental results indicate that plastic deformation is heterogeneous at different length scales and the value of micro, meso and macro strain by different characterization techniques shows a different value. The value of strain determined at the meso and micro length scale from EBSD and X-ray diffraction was negligible, however, the macro-strain as determined from X-ray peak shift was significant. EBSD results showed evidence of profuse \{10\overline{1}2\} \{10\overline{1}0\} contraction twinning in the zinc layer with higher intragranular misorientation in the twin compared to the matrix. It is therefore, inferred that the evolution of higher intergranular (between matrix and twin) strain due to prolific contraction twinning contributes to the failure of zinc layer on galvanized steel.

1. Introduction

Hexagonal close packed (HCP) metals and their alloys find extensive applications in automobile, aerospace and biomedical industries. HCP metals and alloys are characterized with respect to the ideal c/a ratio of 1.633 as less than ideal c/a ratio materials like titanium and zirconium and more than ideal ratio like zinc and cadmium. They undergo deformation by different slip systems namely; \{0002\} \{1120\} basal, \{10\overline{1}0\} \{1120\} prismatic and \{10\overline{1}2\} \{11\overline{2}0\} pyramidal \langle a \rangle slip systems. However, these slip systems do not provide five independent slip systems to accommodate imposed strain according to von Mises criterion. These slip systems are insufficient to provide strain along \langle c \rangle direction and additional mechanisms like \{10\overline{1}2\} \{1\overline{T}23\} pyramidal \langle c+a \rangle and \{10\overline{1}2\} \{011\} extension and \{2\overline{h}\overline{2}\} \{2\overline{h}\overline{3}\} contraction twinning systems are also operative [1]. Twinning, unlike slip causes sudden reorientation and a significant characteristic strain is associated with it. Twinning contributes to strain hardening thereby aiding in ductility but at the same time, sudden reorientation can also cause initiation of cracks in the material at the twin-matrix interface. However, the twin shear (s) for most twin systems in HCP materials is lower (for 10-12 twinning, s = 0.12 for ideal c/a ratio) compared to that in face centre cubic (FCC) materials (s = 0.707) [2]. Hence the twins in HCP materials are wider compared to lenticular twins in FCC materials. This in turn leads to reorientation of large volume of grains to the twinned orientation in hcp materials compared to FCC materials. Reorientation due to twinning can aid in activity of favourable slip system with low critical resolved stress as observed in titanium and zirconium and lead to sufficient ductility. However, in case of brittle HCP materials like magnesium and zinc, reorientation does not lead to easy slip activity and twinning can contribute to failure. In order to decipher the reasons of evolution of the failure, the evolution of stress at different length scale has to be monitored to elucidate the causes of failure. This is achieved by monitoring the evolution of strain at different length scales using X-ray peak shift and broadening, Electron Back Scatter Diffraction (EBSD) and Digital Image Correlation (DIC) techniques. In order to achieve simple stress and strain state, two dimensional polycrystal offer a model material [3]. In the present investigation, strain
evolution at different length scales was monitored in a zinc coating of hot-dip galvanized steel. The X-ray peak broadening of the samples after and prior to tensile test have been compared and the strain evolution was measured at the angstrom scale [4]. EBSD was performed on the deformed sample and strain evolved at the meso-scale was estimated by analyzing the orientation maps obtained [5]. DIC technique provided an estimate of strain at the meso length scale using optical method to measure strain from speckle pattern on the specimen using mathematical correlation analysis from digital images taken during the mechanical test. The sample was prepared by application of a random dot pattern (speckle pattern) then consecutive images are captured during the deformation to understand the change in the sample behaviour and surface characteristics when subjected to incremental loads in the millimeter or continuum scale. Such investigations have been carried out separately but in this work we aim to correlate them together and study the deformation occurring in the zinc coating in galvanized steel sheets.

2. Experimental
Commercially available hot-dip galvanized plain carbon steel sheets of about 0.8 mm thickness were studied in the present investigation. Initial characterizations of the coating were performed on the as received sample by optical microscopy, X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) with an Energy Dispersive Spectroscopy and Electron Back Scatter Diffraction facility. The as-received galvanized sheet was cut into small sections, mounted and fine polished by 5µm alumina slurry followed by 1µm alumina slurry. It was etched with 5 ml HCl in 100ml water for 20 seconds and observations were made by an Zeiss Axioskop 2 MAT microscope equipped with a digital camera Axiocam ERC5s. Rectangular sheet of dimensions 6.8cm x 2cm was cut and a tensile specimen was punched out from it. The tensile specimen was etched with 5ml HCl in 100 ml water and a tensile test was performed with it and the fracture was recorded using a video camera placed over a magnifier. Another set of samples with 100 mm length and 15 mm width were provided with a collar and were coated with a speckle pattern to carry out tensile test using DIC.

Fig. 1: Initial microstructure of the zinc layer on the steel sample (left) and snapshots from video during the tensile test.

3. Results and discussion
The initial characterization of the zinc coating under optical microscope reveals very large grains of Zinc of about 500 micron size (Fig. 1). SEM results indicate that the thickness of the coating was 13.8 ±1.8 µm indicating that the zinc coating on a galvanized steel sheet can be considered as a classical case of a two dimensional microstructure. Cross section Energy Dispersive Spectroscopy analysis indicated variation in zinc content from the surface with 100% Zinc to the centre with 100% iron. It is to be mentioned here that the steel layer and the zinc layer is separated by a layer of complex Fe-Zn intermetallics. The presence of
elemental zinc is confirmed by X-ray diffraction patterns (Fig. 2) that shows only zinc peaks though some minor peaks are also observed. The X-ray diffraction spectra shows a very high intensity (0002) peak compared to the other peaks of Zinc which suggests that the sample has a very strong texture with almost all the grains having their <c> axes pointing out perpendicular to the plane of the sheet. The tensile tested sample showed brittle behaviour (not included in the manuscript) and the engineering stress-strain curve started dropping after yield. It is to be mentioned here that the engineering stress-strain curve is for the entire galvanized sheet and showed ultimate tensile strength of 132 MPa. Multiple tests carried out led to failure near the grip section and design modification to ensure failure in the grip section using a dumbell shape specimen is in progress. Snapshots of the video during the tensile test shown in Fig. 1 indicate intergranular nature of fracture in the zinc layer similar to the one reported in literature [6-7].

In the deformed sample, the intensity of (0002) peak decreased and (10-10) and (10-11) peaks gained prominence as seen in Fig. 2. A quantitative estimate of texture is obtained by plotting the ratio of intensities of different peaks (Table I). The decrease in intensity ratio of 0002 peak to 10-10 peak indicates a transition from basal (c-axis out of plane) to prismatic texture (in-plane c-axis). In addition, there was shift in 0002 peak position at lower angle (Fig. 2 and Table I) indicating presence of Type III tensile residual stress. Thus, there is an evolution of macroscopic strain of 0.0032 in the zinc layer. However, microscopic strain evolution as manifested in terms of peak broadening as seen is negligible. The crystal orientation map obtained from EBSD (Fig. 3a) clearly shows the presence of contraction twins on the surface of the zinc layer. The reorientation of grains due to twinning can explain the change in intensity ratio of different XRD peaks indicating change in crystallographic texture. Different parameters like Minimum Angular Deviation which is deviation of angle between Kikuchi bands from the ideal relationship for a EBSD scan, Local Average Misorientation which is the average misorientation in a grain and Strain contouring which is obtained by considering misorientation between larger regions of microstructure are not significant (Table II) and are in the same range as that of undeformed sample. This indicates that failure is not occurring due to processes at this length scale. The DIC map shows that strain evolution is concentrated in the form of bands perpendicular to the direction of loading and that high strain evolves at the area close to the final failure (Fig. 3b). However, it is the macroscopic tensile stresses which are expected to contribute to failure. This is counter intuitive as compressive stresses are expected normal to the sheet plane during tensile test along the axis. This can be explained on the basis of contraction twinning in the zinc layer due to compressive stresses which is compensated by tensile stress in the matrix and is reflected as shift in the 0002 peak. The incompatibility within the grain and stronger character of the twin boundary ensures that crack propagates in an intergranular manner as shown in Fig. 4. Thus macroscopic stress evolution in the grains contributes to the failure of zinc layer on galvanized steel sample.

Fig. 2: X-ray diffraction spectra of the as received and deformed sample with close view of 0002 peak.
Fig. 3: (a) Crystal Orientation map from the deformed sample showing contraction twinning and (b) DIC image of the deformed sample with RGB corresponding to strain from 0 to 0.18 of von Mises strain.

Fig. 4: Schematic showing the mechanism of failure in the zinc layer on galvanized steel.

Table 1: Texture and microstructural parameters obtained from X-Ray diffraction.

| Parameter | $I_{002} / I_{100}$ | $I_{002} / I_{101}$ | Crystallite size | Micro strain | Macro strain |
|-----------|---------------------|---------------------|------------------|--------------|--------------|
| Initial   | 60.49               | 53.85               | 836.2 nm         | 0.00052      | ---          |
| Deformed  | 6.39                | 20.44               | 491.9 nm         | 0.00070      | 0.0032       |
Table 2: Strain estimation at different length scales.

| Length scale     | Strain       | Technique |
|------------------|--------------|-----------|
| Continuum (mm scale) | 0.18         | DIC       |
| Macro            | 0.0032       | X-Ray     |
| Meso             | LAM: 0.1662° MAD: 0.2699° SC: 0.2699° | EBSD     |
| Micro            | 0.0007       | X-Ray     |

4. Conclusions
To summarize, it can be inferred that the micro-strain evolution in the zinc layer on steel was negligible, the meso-strain was substantial while the macro strain was the most significant. Thus, the failure of zinc coating on galvanized steel is facilitated by evolution of macro strain accompanied with contraction twinning.

Acknowledgments
The authors would like to thank Prof. S. Sangal (MSE IITK) and Prof. P. Venkitnarayanan (ME IITK) for extending various experimental facilities available in their labs. AG thanks IITK SURGE fellowship that made his stay at IITK possible.

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