MSE642A

Microscopy and Microanalysis of Materials

Spiral Microstructure in Eutectic Zn-Mg

Nivedita[1]

[1]Materials Science and Engineering, Indian Institute of Technology, Kanpur, Uttar Pradesh - 208016, India

Correspondence
Email: nivdita@iitk.ac.in

This is an attempt to image the microstructure in eutectic Zn-Mg which appears as intertwined spirals in the cross section. Images the various surfaces of the sample in the bright-field, dark field, DIC, and directional dark-field modes in the optical microscope were obtained. Further, analysis was continued in an SEM. SE images were obtained at various magnifications and compositional analysis was done via EDS.

KEYWORDS

Spiral Microstructure, Eutectic Zn-Mg, Optical Microscopy, SEM

1 | PHASE DIAGRAM AND THEORY OF SPIRAL FORMATION

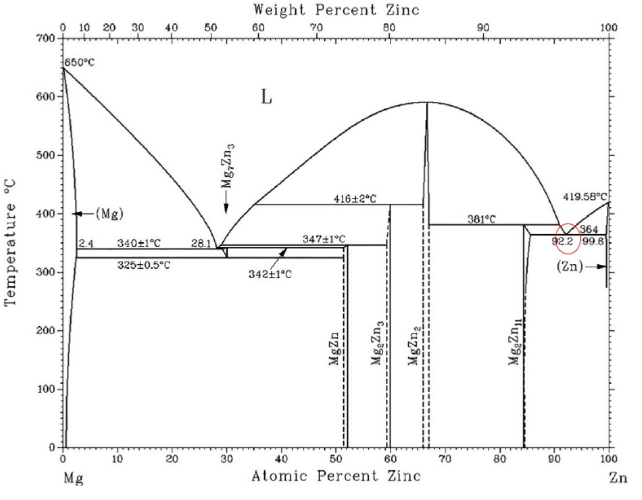


FIGURE 1 Phase Diagram of Zn-Mg (from Lü et al. (2013))

Abbreviations: SEM, Scanning Electron Microscopy; DIC, Differential Interference Contrast; SE, Secondary Electrons; EDS, Energy Dispersive X-ray Spectroscopy

TABLE 1 Zn-Mg Eutectic Composition

Variables	Zinc	Magnesium
Atomic Weight (u)	65.38	24.31
Atomic Percentage (%)	92.2	7.8
Weight Percentage (%)	97.0	3.0
Density ($g \cdot cm^{-3}$)	7.13	1.74
Melting Point (°C)	419.53	650
Boiling Point (°C)	907	1090

A small amount of alloy (0.3g of Mg and 9.7g of Zn) was melted in an induction furnace in a graphite crucible. Three perpendicular Cross sections were prepared for metallographic examination by mechanical polishing and etching with 1% Nital.

1.1 | Spiral Formation

According to Fullman and Wood (1954), a typical eutectic solidification consists of the growth of adjacent rods of the two phases. The composition of liquid adjacent to the rapid-growth directions of each rod is shifted towards the composition of the other phase. As a result, the liquid at the ends of the slower-growing (β)-rod is more"supercooled" with respect to growth of phase-α than is the liquid at the ends of the α-rod. Hence the faster-growing phase tends to grow around the slower-growing phase. Continued growth of the pair of two-dimensional rods would then lead to formation of a spiral. In three-dimensions, the faster-growing phase grows around the slower growing phase in all directions.

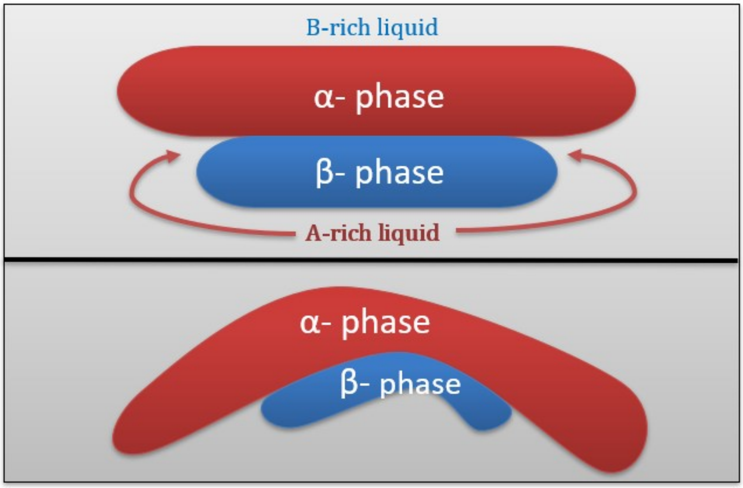


FIGURE 2 Schematic growth of rods during solidification of a hypothetical eutectic alloy to demonstrate initialisation of spiral formation

2 | OPTICAL IMAGING

Optical Microscopy images were obtained of the polished and etched samples in *Olympus BX3M* in various modes.

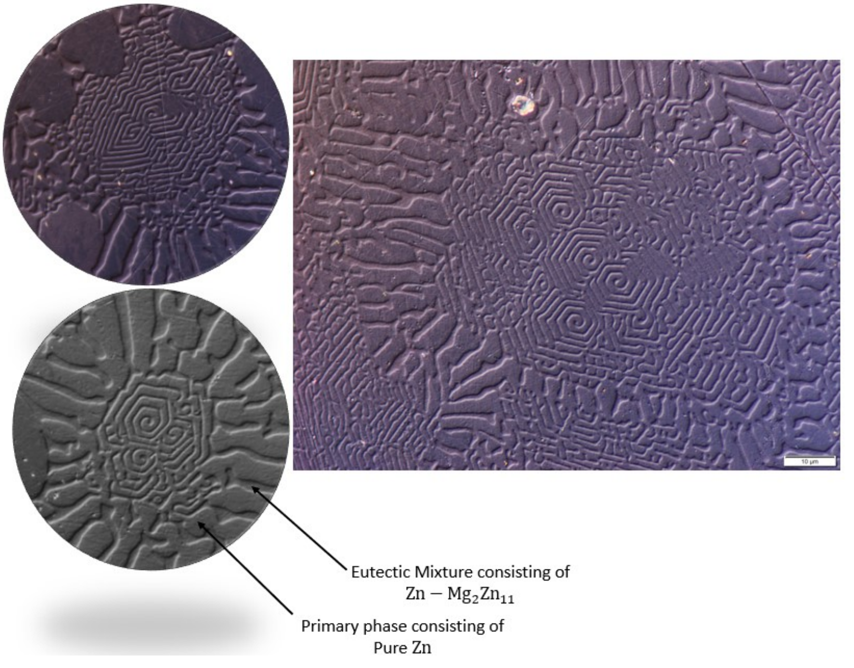


FIGURE 3 Optical micrograph illustrationg Spiral Structures in Eutectic Zinc rich Zn-Mg alloy. The primary phase, pure Zinc and the eutectic mixture of Mg_2Zn_{11} are the two phases.

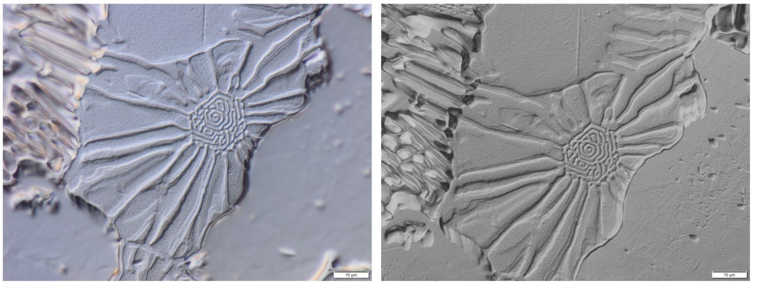


FIGURE 4 A single grain showing the spiral structure in the centre. The images are taken in the DIC mode.

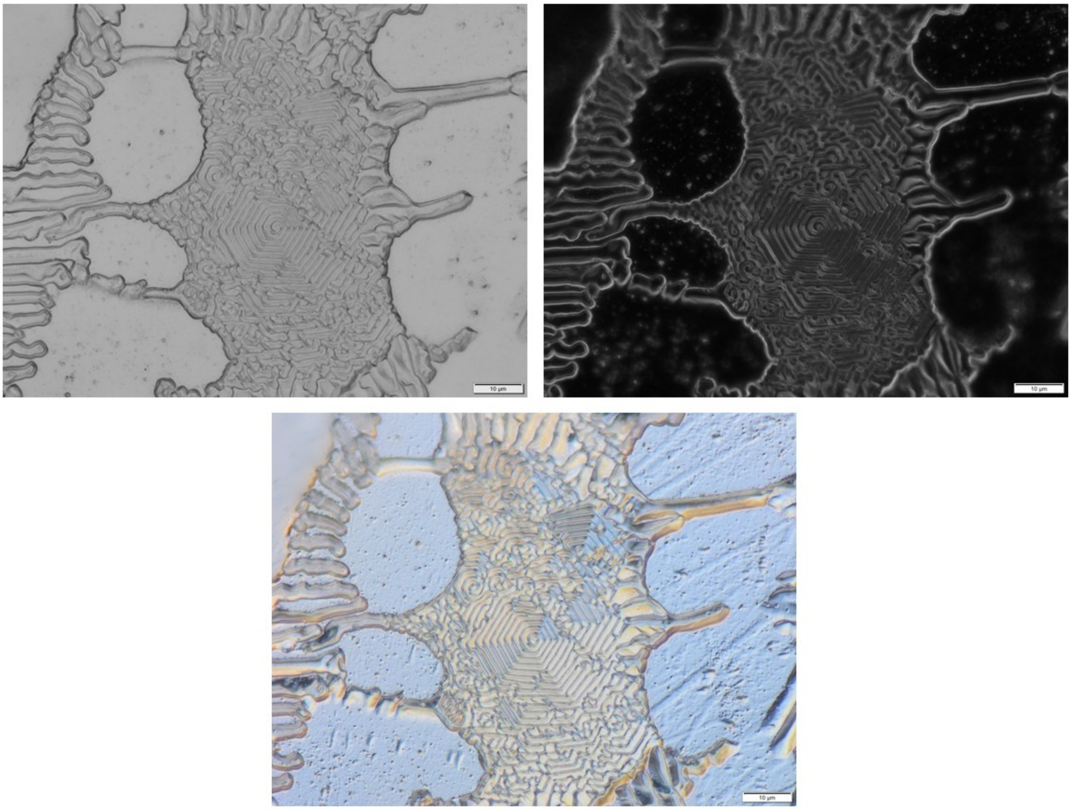


FIGURE 5 Bright field (left top), Dark field (top right) and DIC (centre down) modes showing spiral eutectic.

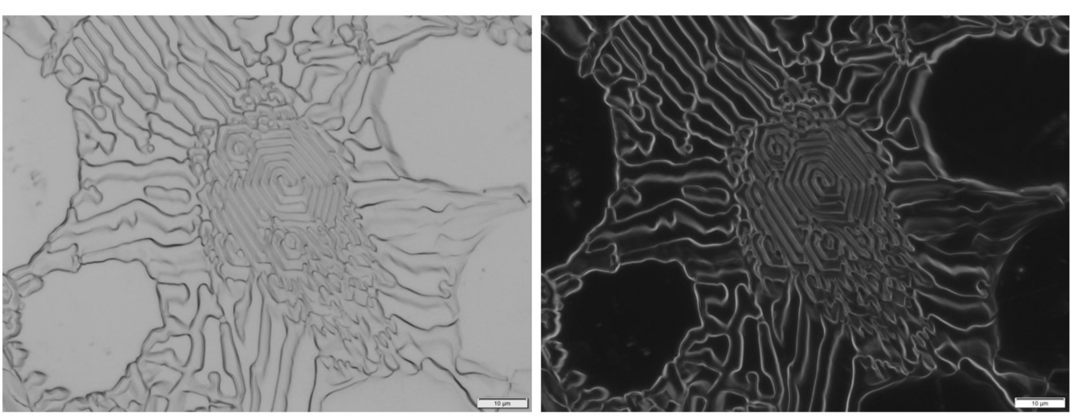


FIGURE 6 Bright Field (left) and Dark Field (right) mode Image. The edges look dark in bright field and bright in dark field

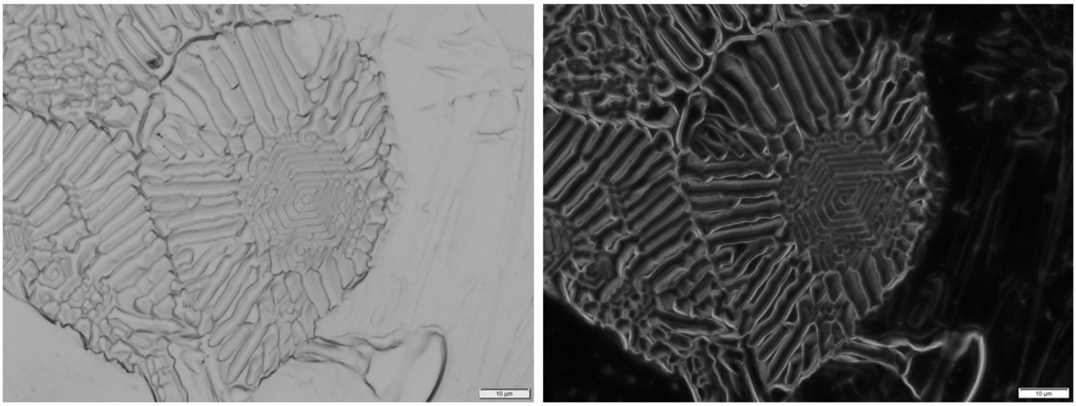


FIGURE 7 Bright Field (left) and Dark Field (right) mode image in a grain.

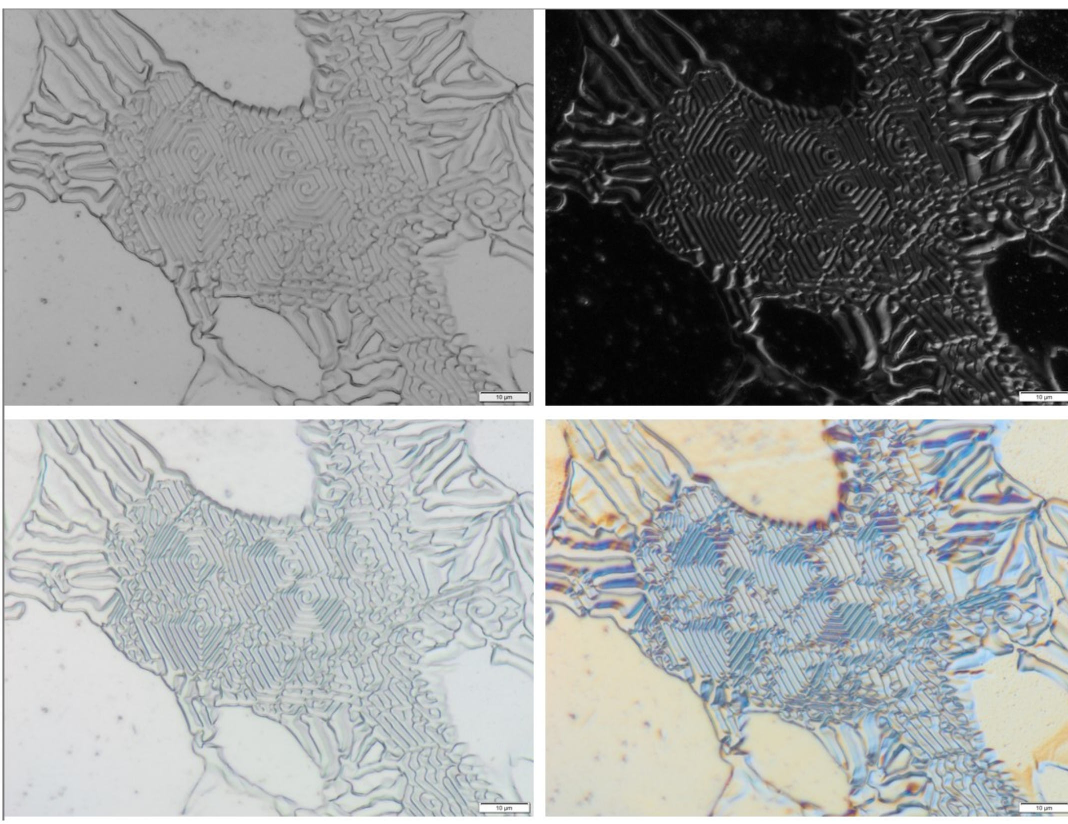


FIGURE 8 Bright field (left top), Directional Dark field (top right) and DIC grey (down left) and DIC coloured (down right) modes showing spiral eutectic structure, with numerous hexagonal spirals impinging on each other, orientations of adjacent spirals can be the same or opposite. In Directional Dark field mode, the incoming beam can be made specific from a specific arc segment of the annulus

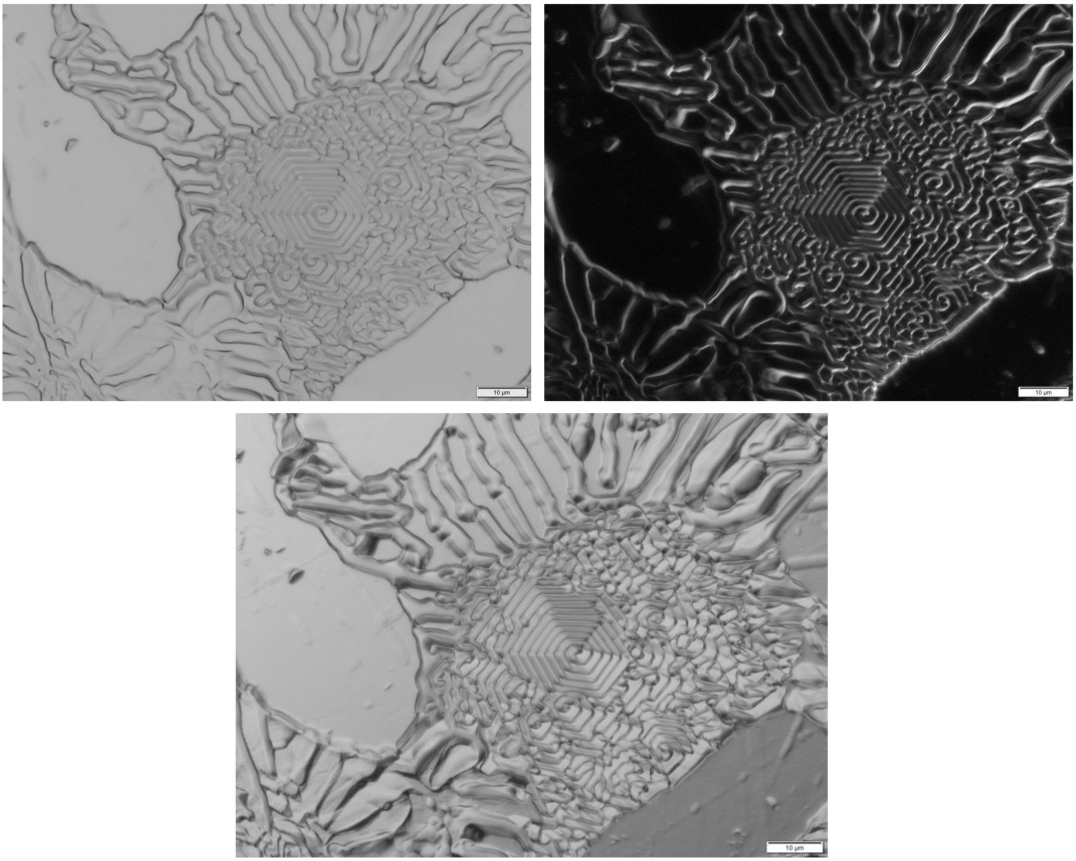


FIGURE 9 Bright field (left top), Directional Dark field (top right) and DIC (center down) modes showing spiral eutectic in another grain.

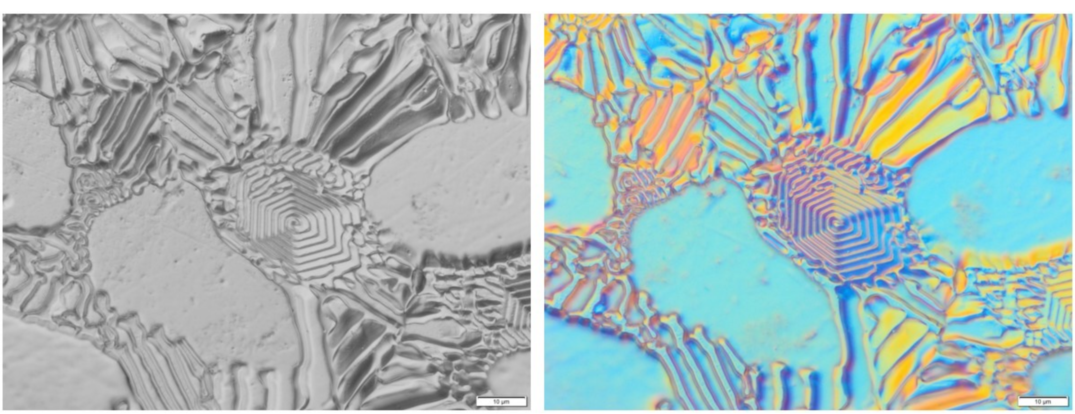


FIGURE 10 DIC grey (left) and coloured (right) clearly showing how single spirals are faceted hexagonal cones.

3 | SEM

Tungsten-Electron Microscope(W-SEM) *JSM-6010LA;JEOL* at ACMS IIT Kanpur was used to obtain images in SE mode as well as compositional analysis was done using EDS.

3.1 | Secondary Electron Images

Imaging with secondary electrons provides information about morphology and surface topography. The contrast is dominated by edge effects. i.e. more secondary electron can leave the sample at edges than in flat areas leading to increased brightness there. SE Images in SEM provide a higher depth of field.

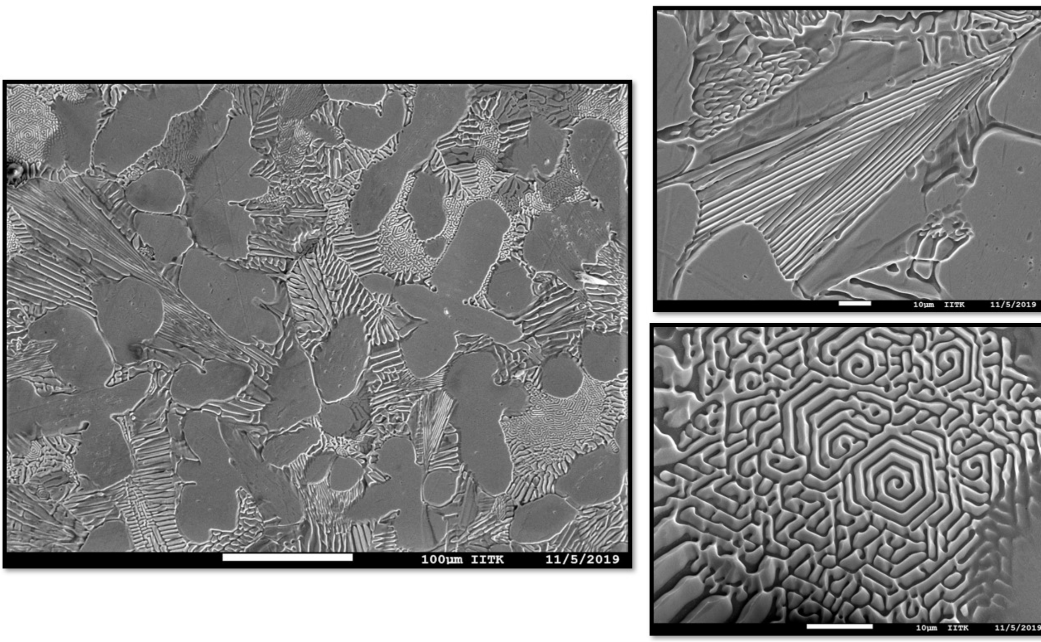


FIGURE 11 Spiral Eutectic Structure showing transversal and axial cross sections of the spiral cones

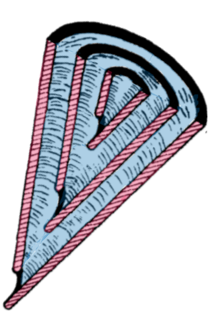


FIGURE 12 Schematic diagram of axial cross section through a spiral cone, Fullman and Wood (1954)

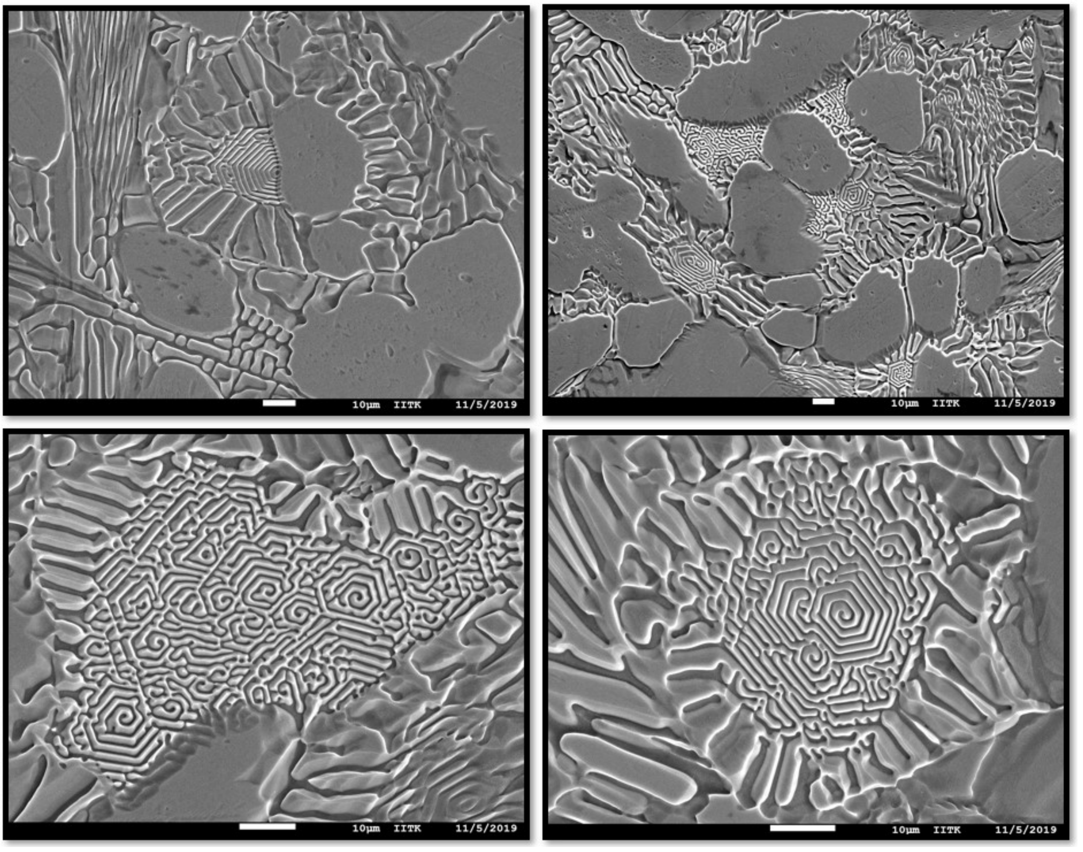


FIGURE 13 Numerous Spirals Impinging each other during Growth

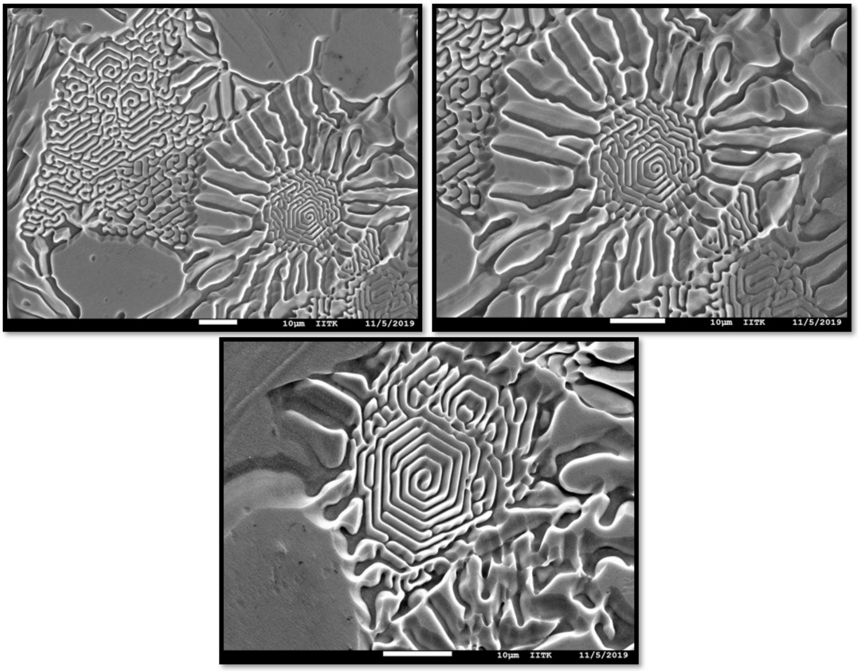


FIGURE 14 Spiral Structures along with standard eutectic microstructure

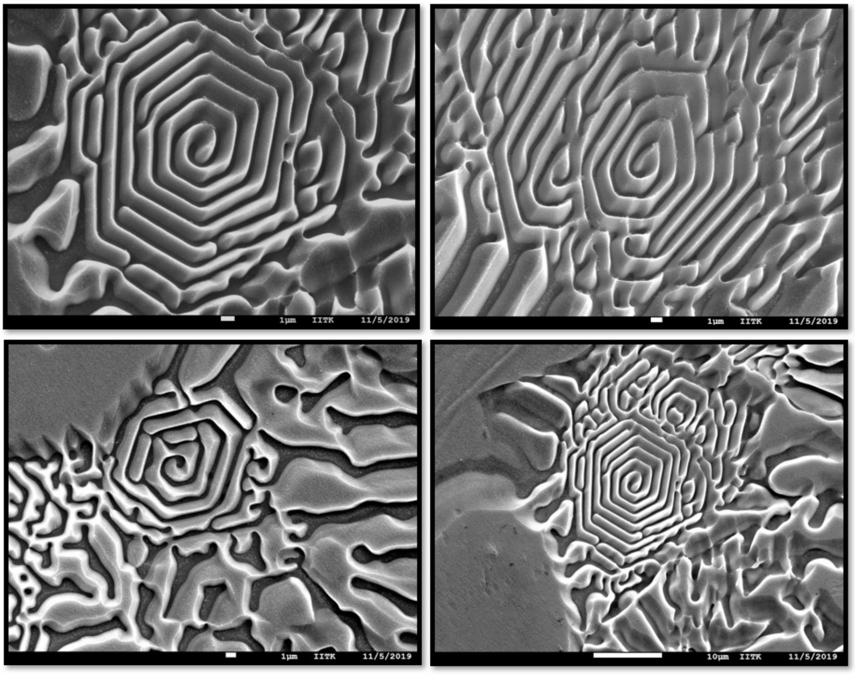


FIGURE 15 Spirals having approximately Regular Hexagonal form

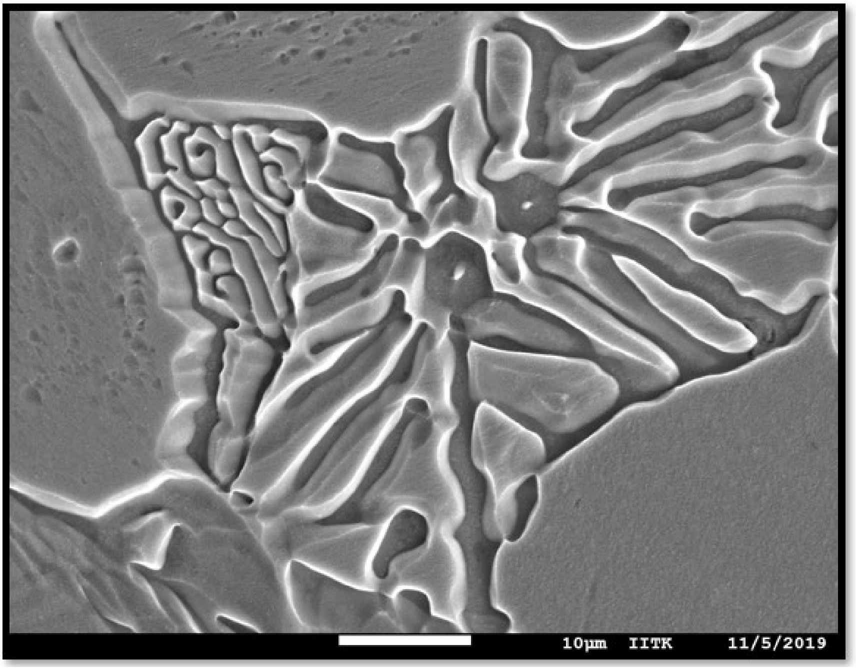


FIGURE 16 Nuclei of the spiral structure, these could also be the centre vertex of double cones.

4 | ENERGY DISPERSIVE X-RAY SPECTROSCOPY

Energy Dispersive X-Ray Spectroscopy (EDS) is a chemical micro-analysis technique used in conjunction with scanning electron microscopy (SEM). The EDS technique detects x-rays emitted from the sample during bombardment by the electron beam to characterize the elemental composition of the analyzed volume.
When the sample is bombarded by the SEM's electron beam, electrons are ejected from the atoms comprising the sample's surface. The resulting electron vacancies are filled by electrons from a higher state, and an x-ray is emitted to balance the energy difference between the two electrons' states. The x-ray energy is characteristic of the element from which it was emitted. The EDS x-ray detector measures the relative abundance of emitted x-rays versus their energy.
The sample x-ray energy values from the EDS spectrum are compared with known characteristic x-ray energy values to determine the presence of an element in the sample.
Quantitative results are obtained from the relative x-ray counts at the characteristic energy levels for the sample constituents.

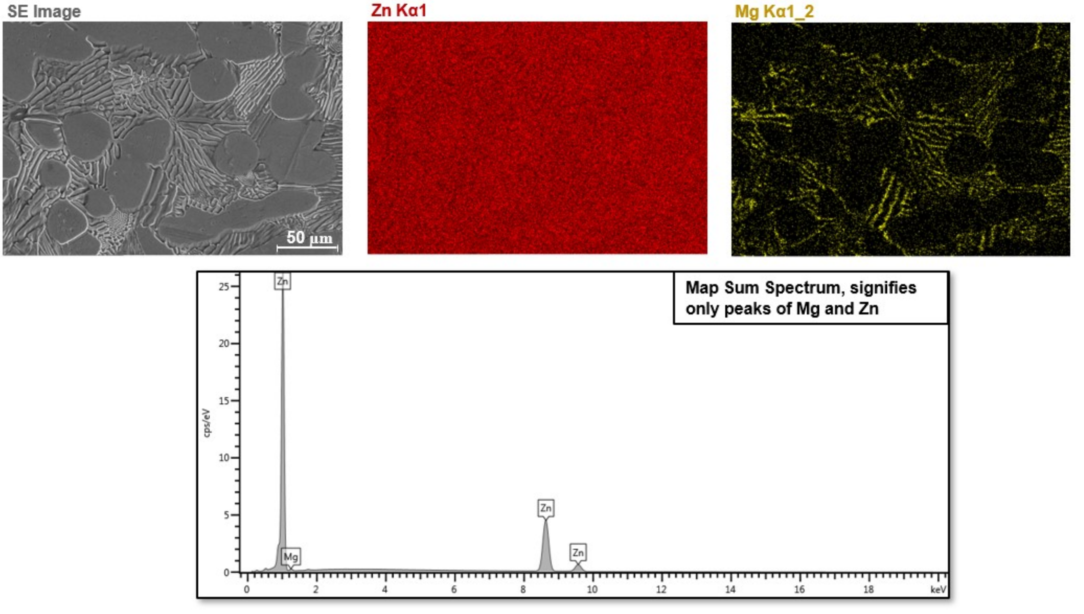
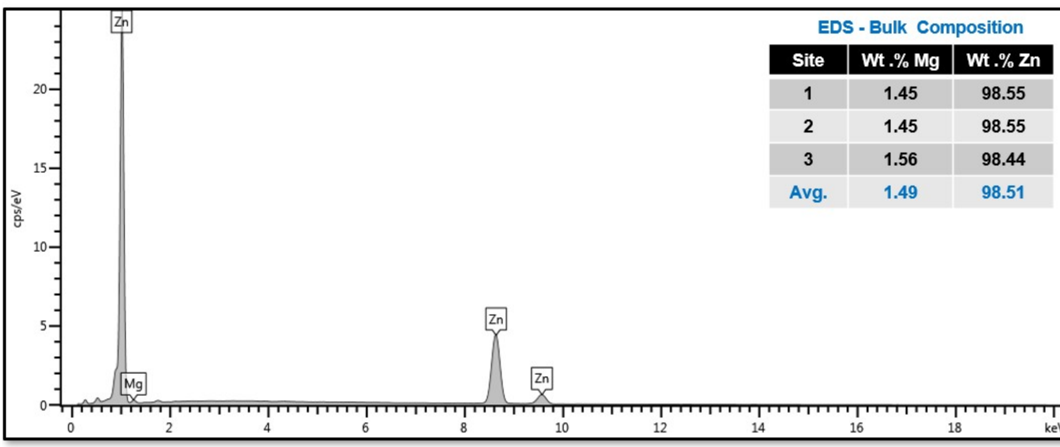


FIGURE 17 Area Map, segregation of of Magnesium to the grooves of the spiral and an approximately uniform Zinc distribution



Site	Wt .% Mg	Wt .% Zn
1	1.45	98.55
2	1.45	98.55
3	1.56	98.44
Avg.	1.49	98.51

FIGURE 18 Bulk Composition of the alloy indicating Zn and Mg peaks

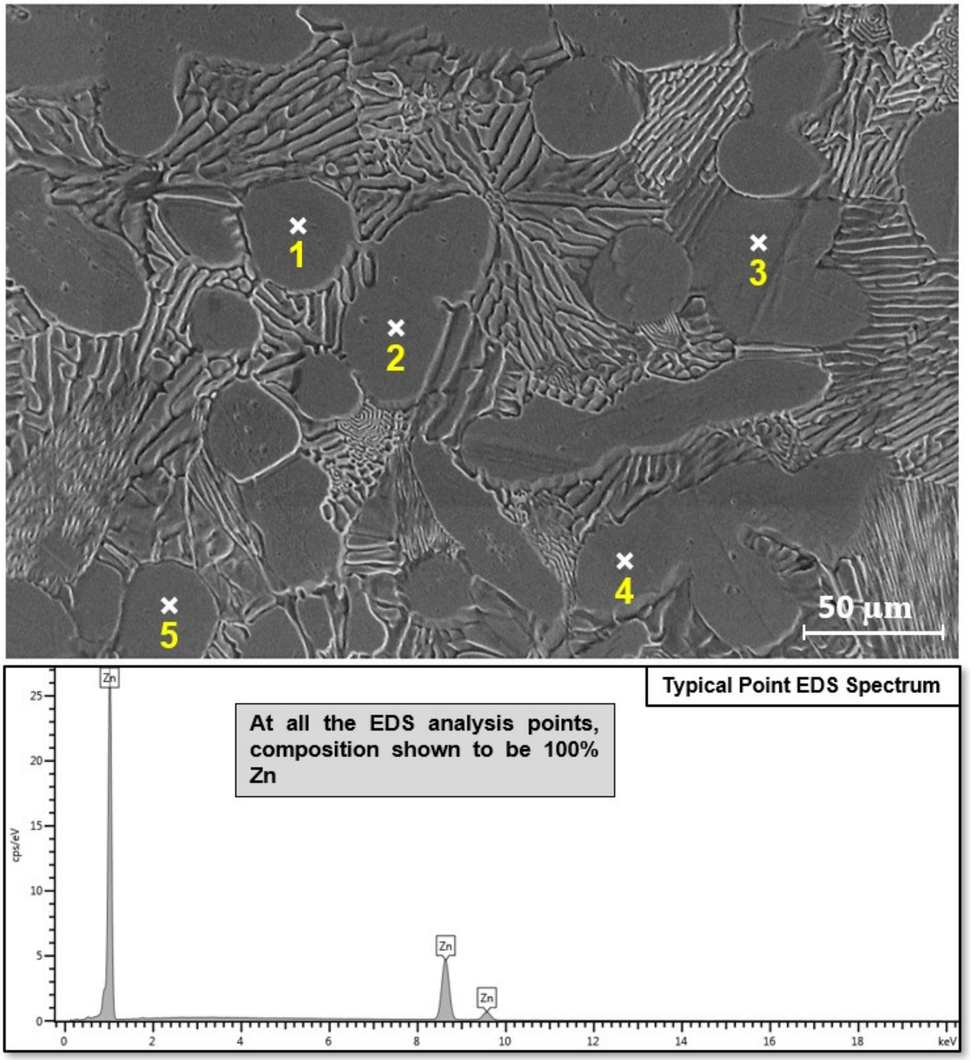


FIGURE 19 EDS point analysis done at spaced points in sample which visibly belonged to a distinct phase, results confirm them to be unalloyed 100% zinc.

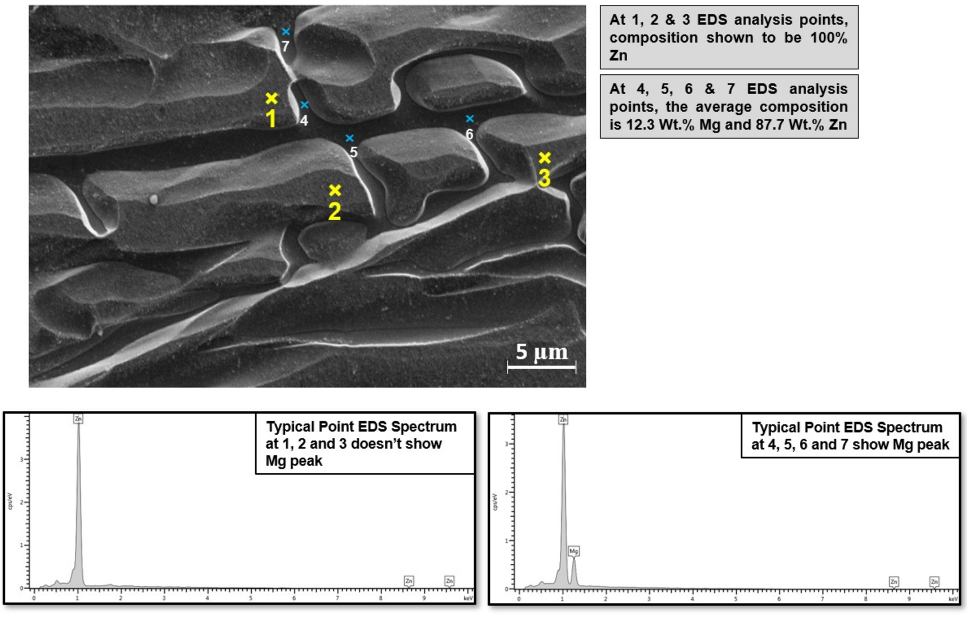


FIGURE 20 EDS point analysis done of the spiral, showing that pure Zn forms the spirals whereas, Mg segregates in the grooves of the spiral.

ACKNOWLEDGEMENTS

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