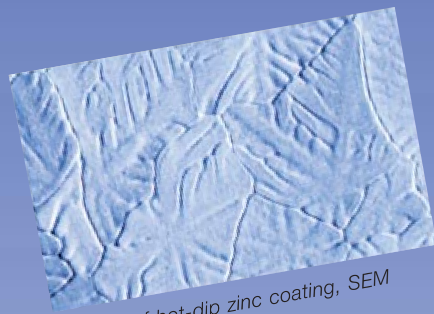
Metallographic preparation of zinc coatings

Struers

Application Notes



Surface of hot-dip zinc coating, SEM

The coating of steel or iron with zinc using various processes is called "galvanizing". Metallic zinc is used for corrosion protection of cast irons, mild steels and low alloy steels. The zinc or zinc alloy cathodically protects the steel surface. In zinc coatings on parts that are exposed to air and water the zinc forms zinc carbonate that additionally protects against corrosion. The coated products are mainly used for exposed automotive applications, household goods, electrical appliances and in the building industry. To increase corrosion protection or the decorative element of galvanized steel sheets, organic coatings such as foils or paint can be applied onto the zinc coating.

Fig.1: Galvalume, etched with 1% Nital, 500x

Fig. 2: Colored building facade of galvanized steel sheets

Although general production control of galvanized sheet steel is done on-line and carried out with physical tests, metallography is an important tool for obtaining a variety of information not achievable by other analytical methods. An understanding of forming, welding and finishing processes of products, product research and development and failure analysis require information only provided by metallographic microstructural analysis.

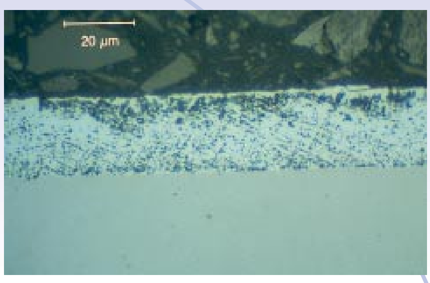
Practical examples: If a zinc coating is too thick problems during welding can arise, if during forming the coating is chafed subsequent painting and consequently corrosion protection can suffer. Therefore metallography plays its part in improving products and advancing coating technology.

The metallography of galvanized products includes thickness measurements of the coatings, microstructure analysis of coatings and basemetal, adhesion checks and failure analysis. Depending on the galvanizing method, zinc coatings vary in hardness and thickness and therefore behave differently during metallographic preparation. In addition, some of the zinc coatings react with water, which makes the preparation particularly difficult.

Difficulties during metallographic preparation

Mounting: *Shrinkage gaps between sample and resin result in stains from water and alcohol, rounded edges and trapped grinding debris. 200 x*

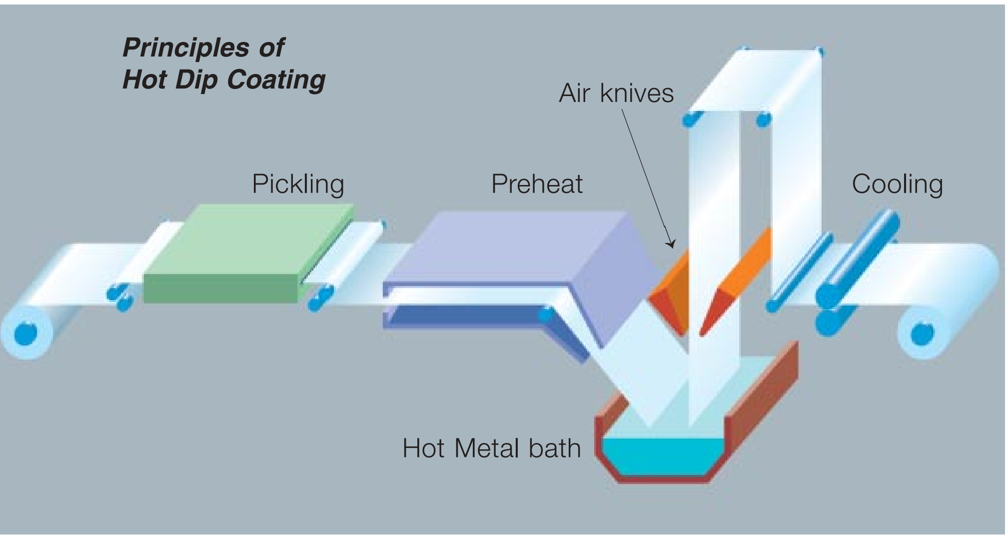
Softness of coatings: *Scratches from grinding and polishing. 500 x*



Reaction with water: *Discoloration and attack of zinc. 1000 x*

Solution: Proper mounting, fine grinding with diamond on rigid discs, using waterfree diamond suspensions and lubricants for polishing and proper cleaning.

Production and applications of zinc coatings



Hot dip
The steel sheet runs through a molten bath of zinc in a continuous process, during which cleaning, temperature, coating thickness etc. are automatically controlled. The coating contains more than 99% zinc and has usually a thickness between 7-15 µm, but can vary according to specifications.
Application: primarily for automotive applications, construction and household goods, such as washing machines.

Galvaneal
After the hot dip the coated sheet can be heat treated, changing the zinc coating into a zinc-iron coating, containing approximately 10% iron. The coating is stronger and has a higher wear resistance and weldability than the plain hot dip coating. On its rough surface paint adheres particularly well, which gives an additional corrosion protection when painted.
Application: primarily pressed automotive components that are coated with organic coatings, outer and inner body panels.

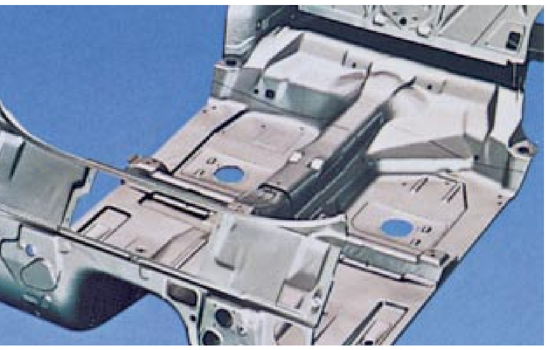


Fig. 3: Pressed floor panel

Galvalume™ 1)
This is a hot dip coating from a bath containing 55% aluminium, 43.4% zinc and 1.6% silicon. This coating has a good heat-oxidation resistance.
Application: construction, mainly facade coverings, also automotive components.

Galfan™ 2)
This is a hot dip coating from a zinc bath containing 5% aluminium and traces of rare earths.

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1) 2) Galvalume™ and Galfan™ are proprietary galvanizing methods. Galvalume™ in Canada is a registered trademark of Dofasco Inc and in the USA Galvalume™ is a registered trademark of BIEC INC. Galfan™ is a registered trademark of the International Lead and Zinc Research Organization in USA.

Fig. 4: Facade of Galvalume sheet with organic coating

The Galfan coating has a fine eutectic structure (see Fig. 10), which is suited for bending, deep drawing and welding, and has an excellent corrosion resistance after painting. Application: construction, automotive and household appliance industry.

Electrogalvanizing
This zinc coating is electrolytically deposited on the steel sheet and produces a very thin, uniform layer of pure zinc. The coating thickness is between 2-6 µm, and it is especially suitable for subsequent painting. A typical application is for car automotive bodies.

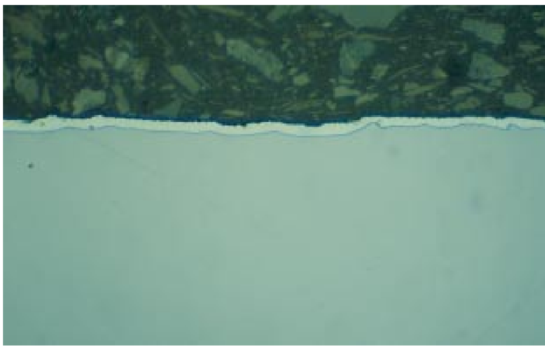

Fig. 5: Electrolytically deposited zinc coating, polished to 1 µm and final cleaning/polish with pure alcohol, 1000 x

Difficulties in the preparation of zinc coatings

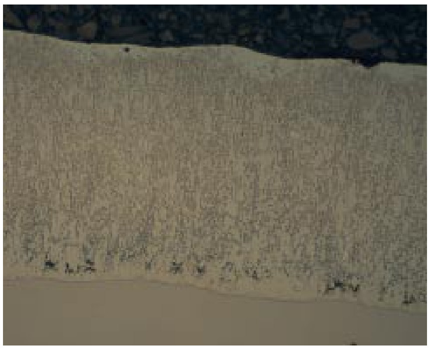

Fig. 6: Post-fabrication zinc coating, etched with 0.5% Nital, with γ phase on the border to the base metal and large δ columns in the zinc matrix, 200 x.

The thickness of zinc coatings of continuous galvanized products is usually specified by weight, g/m² or oz/ft² and can range between 6 µm and more than 20 µm depending on the application of the final product.

For post fabrication hot dip galvanizing of finished parts, such as cast iron construction parts, the finished product is immersed into the molten zinc bath. These coatings are much thicker than the continuously galvanized coatings (Fig. 6).

The main problems of preparing zinc coatings for microscopic observation are:

1. *Shrinkage gaps between mounting resin and coated steel sheets. These gaps make it difficult to clean the samples, especially on clamped sheet packs.*

Mounting zinc coatings constitutes a major problem because the mounting needs to guarantee that the resin adheres properly to the sample material, so that no gaps appear. If zinc coatings are prepared for production control, a conflict arises between number of samples and available time so that compromises in mounting are common. This usually results in a mounting method that produces shrinkage gaps next to the zinc coating, which makes it difficult to clean the specimens and achieve an optimal edge retention. Resulting water and alcohol stains obstruct proper thickness measurement and structure interpretation on the coating.

2. *The softness of zinc and its reaction with water can leave the coatings with scratches, discolored or even etched.*

The purer the zinc of the coating is, the softer and the more water-sensitive it becomes. Therefore, plain hot dip and electrolytically deposited coatings are soft and prone to mechanical deformation and they cannot be cleaned with water (Figs 7 and 8). Ethanol or Isopropanol have to be used for cleaning. Due to the addition of aluminium, respectively heat treatment, Galfan™, Galvalume™ and Galvaneal coatings are harder than plain hot dip and electrolytically deposited coatings. Generally they create fewer problems during polishing and their reaction with water is less vehement.

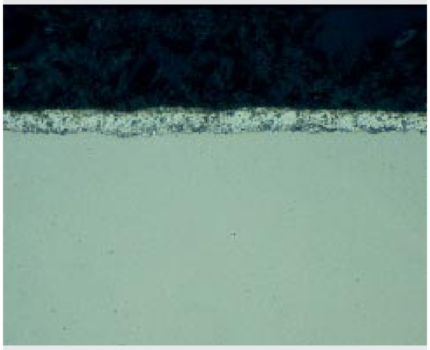

Fig. 7: Hot dip coating cleaned with water, 500 x

Fig. 8: Hot dip coating cleaned with alcohol, 500 x

Recommendations for the preparation of zinc coatings

Cutting steel sheet is not difficult and can be done with appropriate abrasive aluminium oxide wheels. Sometimes sheets are cut with a guillotine or tin snips, which may be sufficient for very thin sheets, but can bend the sheet severely and crack the coating when it is thicker. A subsequent longer grinding time to the undamaged area of the sheet is necessary. Cutting with an abrasive wheel is the more economical alternative in these cases.

Preparation Method

Grinding

Step	PG	FG 1	
Surface	SiC-paper, #320	MD-Largo	
Suspension		DiaPro Allegro-Largo	
Lubricant	Water		
rpm	300	150	
Force [N]	30 per sample	30 per sample	
Time	As needed	4 min.	

Polishing

Step	DP 1	DP 2	OP
Surface	MD-Dac	MD-Dur	OP-Chem
Suspension	DP-Susp. A, 3 µm	DP-Susp. A, 1 µm	
Lubricant	DP-Yellow	DP-Yellow	Ethanol*
rpm	150	150	150
Force [N]	25 per sample	20 per sample	15 per sample
Time	4-6 min.	4-6 min.	15-20 sec.
Comments	**Ethanol denatured with isopropyl alcohol*		

Mounting

The above-mentioned problem of shrinkage gaps between mounting resin and coated specimen can be avoided by degreasing the specimen with acetone before mounting and using proper mounting resins. Slow curing, cold mounting epoxy has a negligible shrinkage and adheres very well to the specimen. Hot compression mounting with a phenolic resin containing carbon fibres (PolyFast) is highly recommended as it leaves no gaps between resin and coating and the resin has the right hardness to keep the samples flat.

For both mounting methods, individual sheet specimens are held upright with plastic clips. Because these sample clips can only hold a few sheets, these methods are too slow for high production quality control laboratories. In these laboratories the most common method for holding coated steel sheets is clamping them in packs: the cut pieces are put into packs and held together by a steel clamp.

This method makes it possible to hold large quantities of sheets, however it does not eliminate the problem of the gaps between the sheets. The clamps are difficult to clean and dry because of the screws and parts they contain, and structure interpretation and thickness measurements are obstructed by alcohol bleeding from the gap. Gluing the sheets together with instant glue and then hot mounting the resulting packs in PolyFast eliminates the gaps and has the advantage of providing a properly mounted, clean sample (see Fig. 9.)

Grinding and polishing

Galvanized steel sheets have traditionally been ground with various grits of silicon carbide paper, followed by two or three diamond polishing steps. Replacing the fine grinding on silicon carbide paper with a special fine grinding disc (MD-Largo), onto which diamond spray or suspension is applied, can now shorten this procedure. MD-Largo also gives excellent edge retention, which is particularly important when observing the coatings at 1000 x. For the subsequent polishing, two diamond polishing steps with 3 µm and 1 µm are sufficient. With this method, the grinding and polishing steps have been reduced from six to four.

Although the different zinc coatings vary in hardness, it is possible to grind and polish them automatically with the same preparation method. When grinding/polishing equipment without an automatic dosing system is used, diamond spray gives excellent results, as it contains no water. For automatic

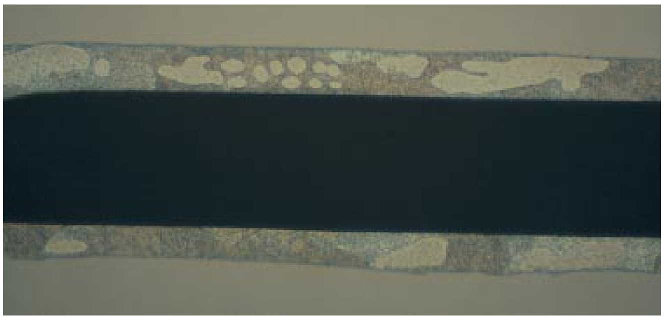

Fig. 9: Galfan coating, etched with 0.5% Nital. Primary dendritic structure, 500 x

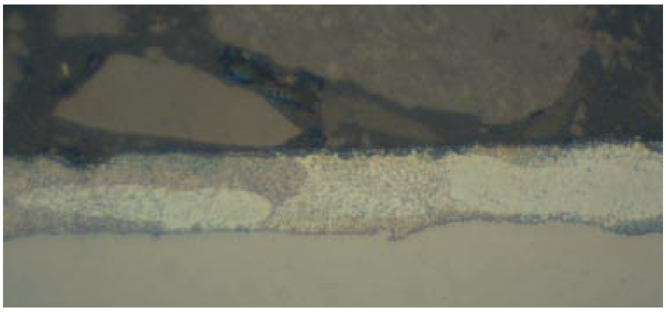

Fig.10: Same coating as Fig. 9, higher magnification shows lamellar eutectic structure, 1000 x

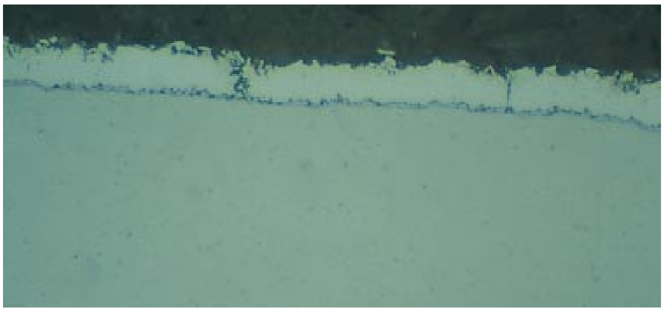

Fig.11: Galvaneal coating etched with 1% Nital, 1000 x

dosing systems, waterfree diamond suspensions and lubricants are necessary to avoid staining of the coatings. Silk cloths keep the samples flat although not completely scratchfree. Polishing with soft, napped cloths should be avoided as it can result in some relief.

Using automatic grinding/polishing equipment with automatic dosing, such as the RotoSystem, has the advantage of giving reliably, very good and reproducible results. The preparation method shown on the previous page, for 30 mm mounted individual samples or multiple samples in holders, has successfully been used for galvanized and electrolytically deposited zinc coatings.

Cleaning

Zinc reacts with water, which makes the cleaning of the samples difficult. During grinding the effect of the reaction with water is negligible, but between diamond polishing steps swabbing and rinsing with ethanol denatured with isopropyl alcohol is recommended, followed by drying with clean compressed air.
For final cleaning, a very brief polish with ethanol denatured with isopropyl alcohol on a MD-Chem polishing cloth, followed by rinsing and drying, has given the best results. If an automatic dosing system is used, a large dose of waterfree lubricant can be programmed to follow after the final seconds of polishing. This will make the subsequent cleaning easier.

Etching and Interpretation

The most common etchant for zinc coatings is a 0.5%-2% alcoholic nitric acid. Etching times are very brief (seconds!) and over-etching occurs very easily, and each coating reacts differently.

Fig. 9 shows a Galfan coating etched with 0.5% Nital. It shows a primary dentritic structure.

Fig.10, Galfan coating as fig. 9, showing a zinc rich solid solution α and a lamellar eutectic structure consisting of α and the aluminium rich phase, β.

Fig.11 shows a Galvaneal etched with a 1% Nital. A thin, iron rich diffusion layer γ shows between steel base and zinc coating. The structure of the coating is zinc-iron with varying concentrations depending on the distance from the base metal. Some of this structure can be revealed using a more sophisticated etchant containing picric and nitric acid. (For the details of this etchant please, see the Struers e-Metalog.)

Summary

To make the preparation of zinc coatings easier and more reproducible, and to improve the images for structure interpretation, the following recommendations for automatic grinding and polishing can be made:

- *Despite varying hardness, all groups of zinc coatings can be automatically prepared using the same method.*
- *The main prerequisite for a successful polish is a good mount without shrinkage gaps between resin and sample.*
- *Fine grinding with diamond on rigid disc gives excellent flatness.*
- *Waterfree diamond suspensions and lubricants keep the zinc coatings from staining.*
- *Silk cloths keep the coatings flat during polishing.*
- *All cleaning steps during polishing have to be carried out with ethanol denatured with isopropyl alcohol.*

The given preparation method can be used as a base for further customising the grinding and polishing procedure in individual laboratories, depending on the variety of coatings to be polished.

Application Notes

Metallographic preparation of zinc coatings

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Credits:
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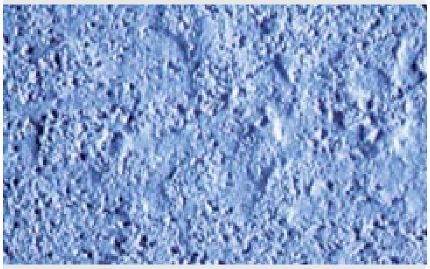

Surface of Galvanealed coating

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