

# STR UCTURE 36



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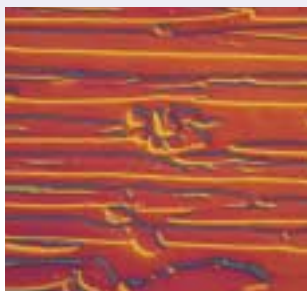
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*Cu58Zn42, sand-cast and cooled very slowly, DIC, 100 x*



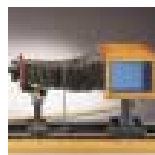
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# Metallographic preparation of zinc coatings

## Introduction

Metallic zinc is used for corrosion protection of cast irons, mild steels and low alloy steels. The coating of steel or iron with zinc using various processes is called "galvanizing". The coated products are mainly used for exposed automotive applications and household appliances.

The metallographic quality control of these products includes thickness measurements of the coatings, structure 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. By using a rigid disc for fine grinding and water-free suspensions and lubricants for polishing, the preparation for these coatings has become easier and less time consuming.

In the following review, the most common methods coating sheet steel with zinc will be briefly described. Some common problems in the preparation of zinc coatings are mentioned, the metallographic preparation method for these coatings will be described, as well as special consumables and/or techniques for overcoming inherent problems of zinc coatings.

## Galvanizing processes

To coat steel sheet the following galvanizing processes are used:

### Hot dip

The sheet runs through a molten bath of zinc in a continuous process, during which cleaning, temperature, coating thickness etc. are automatically controlled. This type of coating contains 99 % zinc and has usually a thickness between 7-15  $\mu\text{m}$ , but can vary according to specifications. Application: primarily for exposed

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, which has a higher strength, wear resistance and weldability than the plain hot dip coating.

Application: primarily pressed automotive components that are additionally coated with organic coating

### <sup>1</sup>Galvalume™

This is a hot dip coating from a bath containing 55 % aluminium, 43.4 % zinc and 1.6 % silicon.

Application: construction, mainly facade coverings, also automotive components

### <sup>2</sup>Galfan™

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

Application: construction, automotive industry and household appliances

The thickness of zinc coatings of continuous galvanized products are usually specified by weight,  $\text{g}/\text{m}^2$  or  $\text{oz}/\text{ft}^2$  and can range between 6  $\mu\text{m}$  and more than 20  $\mu\text{m}$  depending on the application of the final product. For post fabrication hot dip galvanizing of finished parts, the finished product is immersed into the molten zinc bath (example: cast iron parts). These coatings are usually thicker than the continuously galvanized coatings.

### Electrogalvanizing

<sup>1, 2</sup> Both, 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 Organisation in USA

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  $\mu\text{m}$ , and it is especially suitable for subsequent painting. A typical application is for car automotive bodies.

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.

## Preparation problems of zinc coatings

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

1. Gaps between mounting resin and coated steel sheets make the samples difficult to clean, especially clamped sheet packs.

Mounting zinc coatings constitutes a major problem for two reasons: first, the mounting needs to guarantee that the resin adheres properly to the sample material, so that no gaps appear. Second, as most of these zinc coatings are prepared for production control, quantity and time are in conflict and compromises in mounting are common. This usually results in a mounting method that produces gaps next to the zinc coating, which makes it difficult to clean the specimen and to do a proper thickness measurement and structure interpretation on the coating.

2. The softness of zinc and its reaction with water result in scratchy, discoloured or even etched coatings.

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 can not be cleaned with water. Galvaneal and Galvalume™ coatings, which

are harder than plain hot dip and electrolytically deposited coatings, generally create very few problems during polishing.

### Recommendations for the preparation of zinc coatings

#### Cutting

Cutting galvanized steel sheet is not difficult and can be done with a regular abrasive aluminium oxide wheel with an appropriate hardness. 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.

#### 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 is highly recommended as it leaves no gaps between resin and coating and the resin has the right hardness to keep the samples flat.

In both cases, individual sheet specimens are held upright with plastic clips. Because these sample clips can only hold a few sheets, both 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 has several drawbacks:

As the sheets are never completely flat there will always be a gap in the pack somewhere, no matter how tight the clamp is closed. These gaps make it very difficult to clean the samples, which is a constant irritation for the lab staff, because structure interpretation and thickness measurements are obstructed by alcohol bleeding from the gap. The clamps themselves are difficult to clean and dry because of the screws and parts they contain. In addition, the steel clamps have very sharp edges that can shave off the polishing cloths during preparation, making the preparation costly. A combination of the above-mentioned mounting methods, has proven to give good results for clamps. The steel sheets are packed loosely in a small clamp that fits a 30 or 40 mm mount. The clamp with the sheets is dipped briefly into a fast curing cold mounting resin. Then the screws are tightened so that any excess resin is squeezed out between the sheets. By that time the resin starts setting and any excess can be wiped off. This whole clamp is then hot mounted. This results in a very tight pack

without any gaps, in a clean hot mount (Fig. 1). Although this mounting method is more time consuming than plain clamping, it saves time with the faster and better cleaning.

#### 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 with silicon carbide paper with a special fine grinding disc, onto which diamond spray or suspension is applied, can now shorten this procedure. This disc also gives excellent edge retention, which is particularly important when observing the coatings at 1000x. For the subsequent polishing, two diamond polishing steps with 3  $\mu\text{m}$  and 1  $\mu\text{m}$  are sufficient. With this method, the grinding and polishing steps have been reduced from six to four. (On some plain hot dip coatings a 1  $\mu\text{m}$  polish was sufficient, however, as the composition and thickness can vary, this shorter method can not generally be recommended as sufficient for all coatings.) Although the different zinc coatings vary in hardness, it is possible to



Fig. 1: Galvanized sheets in steel clamp and hot mounted





Fig. 2: Hot dip galvanized coating after 2 minutes of 1 µm polish, 500 x



Fig.3: same as Fig.2 after 4 min. of 1 µm polish and final cleaning/polish with pure alcohol, 500x



Fig. 4: Electrolytically deposited zinc coating, polished to 1 µm and final cleaning/polish with pure alcohol, 1000 x

grind and polish them semi-automatically together in the same holder. When grinding/polishing equipment without an automatic dosing system is used, diamond spray gives excellent results, as it contains no water. For automatic dosing systems, water-free diamond suspensions and lubricants are necessary to avoid staining of the coatings. During polishing there is a strong tendency for relief between steel and zinc coating because of the hardness difference of the two materials. Silk cloths keep the samples flat although not completely scratch-free. However, any attempt to use a soft cloth for final diamond polishing results in a pronounced relief between base and coating and between structural elements in some coatings.

The following preparation method for 30 mm mounted individual samples and multiple samples in holders has successfully been used for galvanized and electrolytically deposited

Fig. 5: Galvanealed zinc coating polished to 1 µm, 1000 x



zinc coatings:

PG	SiC-Paper	# 320	until flat	30 N
FG	MD-Largo	9 µm	4 min	30 N
DP1	MD-Dac	3 µm	4-6 min	25 N
DP2	MD-Dur	1 µm	4-6 min	20 N
Cleaning:				
pure alcohol 15-20 sec				10 N

### Cleaning

Zinc reacts with water, which makes the cleaning of the samples difficult. Between the two diamond polishing steps swapping and rinsing with pure alcohol followed by drying with clean compressed air is recommended. For final cleaning, a very brief polish with pure alcohol only, followed by rinsing and drying, has given the best results. Fig. 2, 3, Fig. 4

### Etching

The most common etchant for zinc coatings is a 0.5 -1 % alcoholic nitric acid. Etching times are very brief (seconds!) and overetching occurs very easily because each coating reacts differently. Fig. 5, 6, Fig. 7

### Summary and conclusion

Galvanizing produces a zinc coating that protects steel and iron against corrosion. Manufacturers use different proprietary baths and processes to produce zinc coatings for a variety of applications. Therefore zinc coatings can vary considerably in thickness, hardness and structure. To make metallographic preparation economical and reproducible, polishing trials on semi-automatic

Fig. 6: Same coating as Fig. 5, etched with Nital, 1%, 1000 x



equipment have shown that all coatings can be accommodated with one polishing method. The resulting preparation method uses rigid discs and diamond grinding, which reduces the number of preparation steps and gives quality results for thickness measurements and structure interpretation. The method can be used as a base for further customizing the grinding and polishing procedure in individual laboratories, depending on the variety of coatings to be polished.

### Credits

We wish to thank DOFASCO Inc., and STELCO Inc., Hamilton, Canada, and Fa. Julius Blum GmbH, Höchst, Austria, for generously supplying sample material and related information, and Zinkberatung Ingenieurdienste GmbH, Düsseldorf, Germany, for contributing with general information on zinc coatings.

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 All: Stahl-Informations-Zentrum, Düsseldorf.

Fig. 7: Galvalum polished to 1 µm, etched with Nital, 1%, 500 x



# The History of Struers

125 Years in Words and Pictures with Emphasis on Struers Metallography



*Holger F. Struer,  
founder of H. Struers  
Chemiske Laboratorium,  
1875*

## 1875

Hans Christian Andersen looked out the window of the carriage. For some reason it had stopped in the middle of Skindergade, one of the main streets in his beloved Copenhagen. He saw two workmen handle a large signboard, and Andersen, always curious, tried to read the text. The men put the sign up against the wall, and he could read, "H. Struers Chemiske Laboratorium", a new company to be established, here in the heart of Copenhagen. Yes, it was the young chemist Holger F. Struer, who had seen the need for a laboratory making chemical analysis in the strongly developing "modern society".



*The building at Skindergade 38, the centre of Copenhagen, where Struers is established in 1875*

## 1895

Holger Struer realizes the possibilities of importing chemicals and instruments for the Danish market. Consequently Struers start an import, being the first Danish company to take up this kind of trade.

## 1896

Marius Grubb, a young chemist, is employed. In due course he and his family shall lead Struers Chemiske Laboratorium to the position we know today.

## 1916

Struers is now the biggest company in the laboratory business, but because of illness, H. Struer must sell the company to three leading co-workers, one of these being Marius Grubb.

## 1917

All import is stopped because of the First World War and Struers makes the first product of own make, a thermometer.

## 1919

Struers obtain the Danish representation of the Austrian company, Reichert, one of the leading microscope suppliers. Reichert is a specialist in metallographic microscopes and for this reason Struers' interest for metallographic analysis is aroused.

## 1928

Kjeld T. Grubb, M. Sc. in chemistry, son of M. Grubb, is employed. He shall later be the driving force behind Struers' metallographic development. In these years own manufacture of products is slowly increasing, and already before the start of the second World War in 1939, a few products are exported.

*"Handmade"  
thermometer,  
produced during the  
period of the First  
World War, 1914-18*



## 1931

Marius Grubb buys out his partners and becomes the sole owner of Struers.

## 1943

Eggert Knuth-Winterfeldt, manager of the materials laboratory in a large Danish industrial company (DISA), contacts Struers with the idea of an instrument for electrolytical polishing. Struers develops and produces an apparatus, Micropol, which is launched in 1944. Struers official "metallographic birthday" is November 15<sup>th</sup>, 1943.

*Micropol 1943*

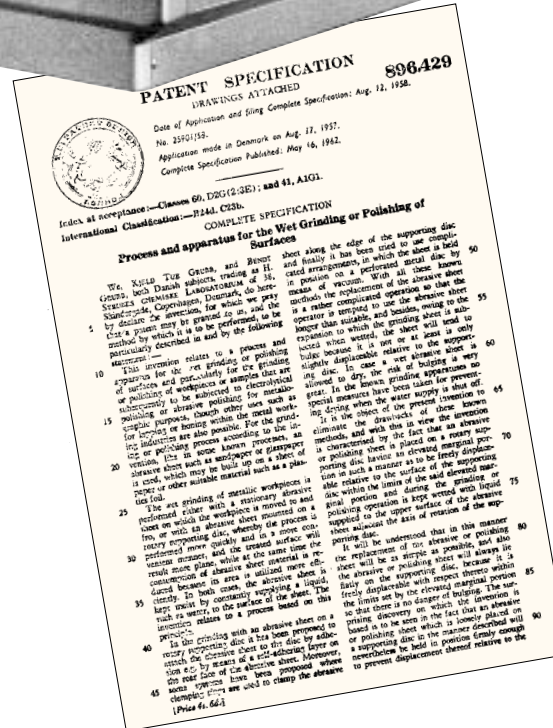


*Microscope for  
metallographic analysis  
made by C. Reichert, Vienna,  
1920*





Dispenser, apparatus for exact dosing of liquids, one of the most important of the "chemical line" of products, 1952



DP-4, the world's first polishing apparatus only for diamond polishing, shown with sample mover PdM-44, 1958



Prestopress, the world's first mounting press with built-in heating and water cooling, 1964

**1949**

Knuth Winterfeldt has been working on the development of a new apparatus, and based on a patent, DISA manufactures Disa Electropol, Struers being the sole distributor worldwide.

**1951**

The production of chemical apparatus, started before the war, has grown, and "Struers Apparatus Factory" is established. In the beginning the production is only chemical apparatus, but over the years, metallographic equipment takes over.

Knuth-Rotor for hand grinding of metallographic samples, 1957. "The Knuth - Rotor-Principle" patented in 1962

**1956**

Disa Electropol was a success in the industrial markets, but Struers realized that electrolytical polishing could not stand alone, mechanical preparation had to be developed. Two patents are registered, one covering diamond polishing, the DP-Method, the other, Knuth-Rotor, a machine for grinding with SiC paper.

**1961**

Struers has got a foothold on the German market and a daughter company is established in Düsseldorf.

**1948**

Marius Grubb dies and his two sons, Kjeld T. Grubb and Bendt Grubb take over.



Kjeld T. Grubb, second generation of the Grubb family as owner of Struers.



Discotom, cut-off machine, showing completely new ways of design in cutting machines, 1965

Lectropol, the first industrial electropolisher developed and made by Struers, 1968







*Abraplan for grinding and Abrapol for polishing, first machines in the world for "production" of materialographic samples, 1973*

**1963**

A strategy is worked out:

- 1) Struers shall develop a complete product program for materialographic preparation.
- 2) All Struers products shall be advanced compared to the "standard" in the market.
- 3) All product development shall take place in close cooperation with the users.

**1965**

Discotom, a cut-off machine, and Prestopress, a mounting press, are launched.

**1968**

Lectropol, Struers own electro-polisher is launched.

**1971**

Tenupol, an apparatus for thinning of specimens for TEM is brought on the market. Metalog, Struers' first complete catalogue, also a "teaching book", is supplied free of charge to all metallographers.

**1973**

Stuers has realized the importance of automatic preparation, and already in 1970 a seminar is arranged with the leading Swedish metallographers. Struers puts forward the question: "What do you want for automatic preparation"? The result was Abraplan and Abrapol, machines for "production of metallographic specimens". Also in 1973 Planopol/Pedemax, tabletop machines for automatic preparation were launched. The same year, Prontopress, the first



*Prontopress, the world's first full-automatic mounting press, 1973*



*Struers Inc., daughter company in Strongsville, (Cleveland), Ohio, USA, 1976*



*Magnum, the world's first full-automatic cut-off machine for materialographic samples, 1978*

fully automatic mounting press was brought to the market.

**1976**

Struers Inc., an American daughter company is established in Cleveland, Ohio, this ensuring an efficient sales of Struers products in USA. In a few years, more than 100 Abrasystems are sold.

**1977**

Magnum, the world's first fully automatic cut-off machine is launched.

**1981**

Structure, Struers journal on materialography is established. Translated into three languages, it is the most read journal covering topics on materialographic preparation.

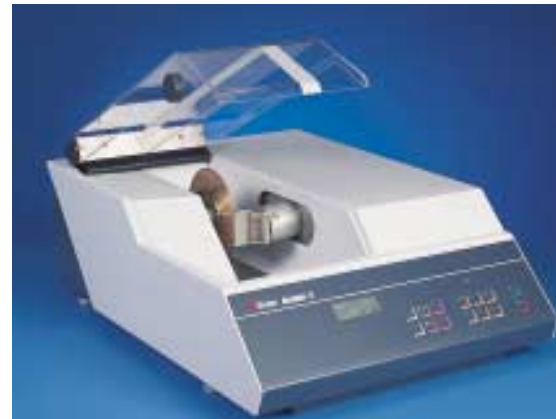


*Abramatic, the world's first microprocessor controlled grinder/polisher for materialographic samples, 1983*





MAPS, Modular Automatic Polishing System for full-automatic preparation of large samples in high volumes, 1991



Accutom-5, a totally new concept in materiallographic precision cutters, 1994



Prepamatic, the world's first grinding/polishing machine with full-automatic function of all preparation steps, cleaning and drying, 1986

**1983**  
Abramatic, the world's first metallographic machine with a microprocessor. Also diamond suspension is introduced, completing the "fully automatic" addition of abrasives during the preparation process.

**1985**  
Prepamatic, the world's first fully automatic grinding/polishing machine is launched, setting new standards for reproducibility and preparation of difficult materials.

**1987**  
Exotom, the world's first automatic cut-off machine with oscillating cutting, making cutting of the most difficult materials possible.

**1989**  
The "metallographic part" of Struers now being as big as the domestic, "chemical" part, the company is divisionized in Struers Tech (materialography) and Struers Chem (import of chemicals and instruments to

the Danish market). Through the 1980's, daughter companies were established in Great Britain, France, Austria, Sweden and Japan. In 1988 the company Logitech, in Glasgow, Scotland, is taken over.

**1991**  
MAPS, a system for fully automatic preparation of large specimens is launched.

**1992**  
Metalog Guide is published, distributed to "all metallographers of the world". Until now translated into nine languages and printed in more than 100.000 copies. Struers is taken

over by the Danish company, Radiometer, and Struers Tech is established as a daughter company named Struers A/S.



Exotom, the world's first automatic cut-off machine for materialographic samples using an oscillating cut-off wheel, 1987



MD-System, a line of grinding and polishing discs for magnetic fixation, totally changing the preparation process, 1995



*LectroPol-5, electrolytic polisher with read-out of current-density curves. A long development has taken place since Micropol, 1996*

**1994**

Accutom-5, precision cut-off machine, is launched, setting new standards in precision cutting.

**1995**

Through analysis of the total preparation process, it was evident that using SiC grinding paper was a "weak point". For this purpose Struers develops and launches the MD-System for grinding with composite discs with magnetic fixation.

**1996**

Struers is certified under ISO 9001. LectroPol-5, the world's first electrolytic polisher showing the voltage/amperage curve, before the start of the process. A long development has taken place since the first Micropol in 1944.

**1998**

The capital investment company, EQT, takes over Struers A/S and Struers is established as an independent company including daughter companies in USA, Japan, Germany, UK and France.

**1999**

Exotom-100, an automatic cut-off machine for very large workpieces, is launched.



*Exotom-100, cut-off machine with three different cutting modes for cutting of large workpieces of the most difficult materials, 1999*



*Duramin, automatic Vickers hardness tester in 4 different models, 2000*

**2000**

TenuPol-5, a new model of the famous electrolytic thinner for TEM samples, is launched. Also AbraPol-10 is brought to the market, a new model of the "Workhorse", being used in laboratories with a heavy workload, all over the world.

2000 is also the year, where Struers diversified into hardness testing, launching the micro-hardness tester, Duramin.

And November 15. Struers' 125 years birthday, of which 57 years has been as partner for the metallographers of the world.



*AbraPol-10, grinding/polishing machine for high volume preparation, 2000*



# Fabrication of **Ultrathin Semiconductors** on **Insulator Structures** by **Materials Integration** Techniques

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## **Abstract**

This article describes thinning processes and characterization techniques employed in the fabrication and assessment of a GaAs-on-insulator structure for which the GaAs layer is 10 nm thick and possesses a surface morphology suitable for subsequent heteroepitaxial growth. The thinning methods employed to obtain the structure are described. Spectrally-resolved ellipsometry and high-resolution x-ray reflectivity (HRXR) measurements are performed and compared to model calculations to assess the accuracy of the structure determination. Atomic force microscopy (AFM) is employed to develop and assess final thinning methods that result in 10nm GaAs-on-insulator structures with surface morphologies that are characterized by a 0.3 nm r.m.s. roughness. The materials and dimensions of the structure are chosen so as to be relevant to fabrication of a substrate intended to impact the relaxation of a subsequently grown highly mismatched film.

## **Introduction**

There has been recent interest in developing alternative substrate structures for use in lattice mismatched heteroepitaxial growth.<sup>1,2,3,4,5,6,7,8,9</sup> The strain within a mismatched heterostructure is partitioned between the substrate and the film according to the inverse of their relative thickness. Mismatched heteroepitaxy on a conventional thick substrate thus results in the entire mismatch-derived strain being initially accommodated in the thin epitaxial film and not in the very thick substrate. For films grown beyond a critical thickness, misfit dislocations are introduced and thread-

ing dislocations propagate into the growing mismatched film<sup>10,11</sup>. These dislocations in the film can negatively impact the performance of devices fabricated from the heterostructure. As the "substrate" thickness is reduced to zero, however, the strain in the film is readily decreased. This concept underlies the multiple approaches to making compliant substrates. A compliant substrate consists of a thin 'free-standing' template layer that serves as the substrate for growth. One approach to the formation of such a thin substrate, or "template layer", is the bonding of this template to a "handle wafer" through a compliant medium.

Two structural characteristics that impact the efficacy of the compliant substrate during subsequent mismatched epitaxial growth are the thickness of the substrate (or "template layer") and the bonding media employed to interface the template to a mechanical host (or "handle wafer"). The efficacy and mechanism of strain relaxation of different compliant substrate structures are not completely understood and are areas of active research.<sup>2,6,7,8,9,12</sup> It is clear, however, that the surface of the template layer should have a morphology suitable for epitaxial growth (structurally smooth and free of chemical impurities) and that the template layer should be as thin as possible to maximize its compliance. The technological challenges in realizing the structure increase as the thickness of the template layer decreases.

In the present article, we describe the thinning techniques employed to fabricate 10 nm GaAs template layers by thinning an etchstop structure bonded via a borosilicate glass to a handle wafer. The accurate thinning of the bonded structure is difficult since it requires the removal of hundreds of microns of material in a

manner that leaves only 10 nm of material intact and with a surface morphology suitable for epitaxial growth.

Direct characterization of the template layer thickness over large areas can be accomplished by high resolution x-ray reflectivity (HRXR) as well as spectrally-resolved ellipsometry. AFM measurements of the surface morphology provide an independent measure of aspects of the substrate surface that may have only a subtle effect on the ellipsometry and HRXR data but a large effect on the quality of subsequently grown films. HRXR, AFM, and spectrally-resolved ellipsometry experiments therefore represent a battery of substrate characterization methods on which interpretation of the compliant substrate's effect on subsequently grown mismatched films can be based.

## **Experiments**

A 228.5 nm film of 30 mole% B<sub>2</sub>O<sub>3</sub> borosilicate glass (BSG), (B<sub>2</sub>O<sub>3</sub>)<sub>0.3</sub>(SiO<sub>2</sub>)<sub>0.7</sub>, was deposited onto a GaAs handle wafer. The BSG-coated GaAs handle wafer was then bonded to the etch-stop structure. The etch-stop structure employed in the present study was a heterostructure consisting of 2 mm of Al<sub>0.70</sub>Ga<sub>0.30</sub>As; 1 mm of GaAs; 0.25 mm of Al<sub>0.70</sub>Ga<sub>0.30</sub>As; and 10 nm of GaAs grown on a GaAs wafer by CVD. This double etch-stop was employed with an HF-based selective etch to remove Al<sub>x</sub>Ga<sub>1-x</sub>As<sup>13</sup> and an NH<sub>4</sub>OH:H<sub>2</sub>O<sub>2</sub>-based selective etch to remove GaAs.<sup>14</sup> Figure 1 is a schematic of the structure and thinning process flow. The bulk of the GaAs substrate underlying the etch-stop structure can be removed by mechanical lapping to within ~30 microns of the etch-stop layer. The remaining GaAs substrate is then removed using an H<sub>2</sub>O<sub>2</sub>:NH<sub>4</sub>OH:H<sub>2</sub>O solution in a 30:1:72 by weight ratio. The me-



chanical thinning of the bonded structure must be sufficiently precise so as to allow the etch to stop at the first 2 mm etch-stop over the entire sample surface.

The precision of the lapping equipment described below allows the etch to stop at the first etch-stop over a 4" diameter wafer. The sample to be thinned is secured to a Logitech PM6 polishing jig (right figure) by means of a vacuum chuck for thinning on the Logitech PM5 autolap lapping and polishing machine (left figure) for gross removal of material by an abrasive slurry. A sample monitor used in conjunction with the lapping machine allows one to automate the cessation of the lapping process at a predetermined thickness with a precision of 2  $\mu\text{m}$ . This tool has the ability to automatically monitor and control the flatness of the lapping plate during thinning. This feature is critical for reproducibly thinning flat 4" wafers without inducing convexity or concavity at greater than the 4  $\mu\text{m}$  level.

The thinning operation employed to remove the bulk of the GaAs substrate need not result in a polished finish. The GaAs/AlGaAs selectivity of the peroxide/ammonium

hydroxide etch is sufficient so that a lapped finish on the remaining GaAs can be tolerated. The primary requirement on the substrate removal operation prior to the hydrogen peroxide/ammonium hydroxide etch is to leave a uniform thickness of GaAs material over the lateral dimension of the bonded wafer. Precision lapping of the bonded structure to within 40  $\mu\text{m}$  of the bonded interface using the above equipment in conjunction with a 3  $\mu\text{m}$  alumina abrasive slurry results in a structure for which the remaining GaAs substrate material can be completely removed upon a 10 to 15 minute exposure to the  $\text{H}_2\text{O}_2:\text{NH}_4\text{OH}:\text{H}_2\text{O}$  solution.

The 2 mm  $\text{Al}_{0.70}\text{Ga}_{0.30}\text{As}$  etch stop is then removed by exposure to a static HF solution consisting of 1:10 HF: $\text{H}_2\text{O}$  by weight for two minutes at room temperature under ambient laboratory lighting. The intermediary 1mm GaAs layer is subsequently removed by submersion in a static  $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$  solution of the same proportions used for the first etching step. This etch is continued until color changes are observed (af-

ter ~30 seconds) that signify the exposure and oxidation of the final  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  etch-stop. The 10 nm GaAs-on-insulator structure is realized by removing the final 0.25 mm  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  etch-stop through submersion in a static 1:10 HF:  $\text{H}_2\text{O}$  solution for 12 seconds. The sample is then characterized by spectrally-resolved ellipsometry, HRXR, and AFM.

Spectrally-resolved ellipsometry data were taken with light impinging on the sample at an angle of 69°. Data were recorded for wavelengths between 450 nm and 750 nm in 10 nm intervals. The data presented here is the average of twelve measurements. The sample was placed in the instrument, 4 measurements were taken, the sample was removed, rotated by ~45 degrees, and reinserted in the instrument for 4 additional measurements. The sample was removed again, rotated by another 45 degrees, and reinserted in the instrument for the final 4 measurements. The vertical error bars on the data points represent one standard deviation from the average value. The horizontal error bars reflect the accuracy of the wavelength assignment for each data point. The optical constants employed for modeling the ellipsometry data were obtained from a commercial ellipsometry company.<sup>15</sup>

X-ray reflectivity measurements of the resulting structure employed a Bede D<sup>3</sup> diffractometer and CuK $\alpha$  radiation from a rotating anode generator operating at 50 kV and 300 mW. The beam was conditioned by a double bounce Si 110 monochromator in the (+/-) configuration before impinging on the sample. The intensity of the specularly reflected x-ray beam was measured from 0° to more than 2°. The data were collected by rotating the sample in steps of 10 arcseconds counting for 5 secs/step. The reflectivity data was interpreted in terms of the thickness of the compliant sub-

Logitech PM5 autolap lapping and polishing machine



Logitech PM6 polishing jig



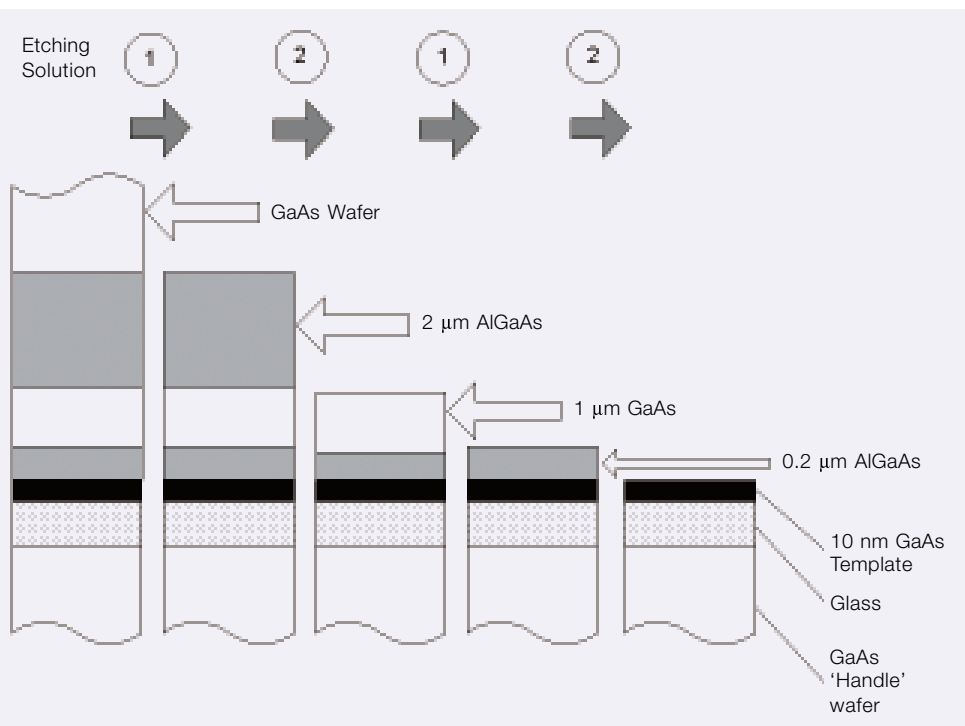


Figure 1: A schematic of the structure evolution and thinning process flow. Etching solution 1 consists of the  $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$  (30:1:72 by weight) and etching solution 2 is  $\text{HF}:\text{H}_2\text{O}$  (1:10 by volume). Precise mechanical lapping of the structure to within  $30\ \mu\text{m}$  of the interface prior to etching allows the formation of the structure over a  $4''$  wafer

taneously regressing on the native oxide and template layer, since each gives rise to qualitatively different effects in the ellipsometric data. From comparison of the data and calculations, we believe that the experiment measures the thickness of the GaAs layer to be 12 nm with a precision greater than 1 nm. The absolute accuracy of the measurement can be assessed through comparison of these data to high-resolution x-ray reflectometry data. The high-resolution x-ray reflectivity data are shown in Figure 4. The experimental data are compared to a model calculation of the x-ray reflectivity behavior of an ultrathin GaAs-

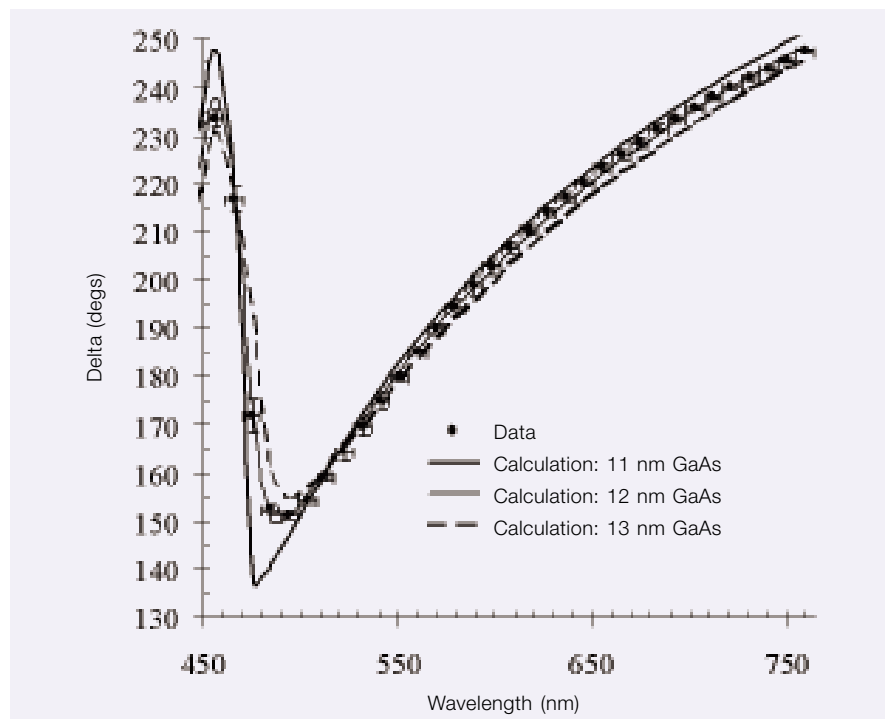
strate's constituent layers using a commercially available simulation package.<sup>16</sup> AFM investigations of the surface morphology employed a Digital Nanoscope III equipped with a silicon nitride tip having a spring constant of 0.12 N/m in contact mode with a set-point that resulted in a contact force of approximately 23 nN.

### Results and Interpretation

The data from spectrally-resolved ellipsometry measurements are shown in Figure 2. In the figure the vertical axis is delta,  $D$ , the phase difference between the reflected p-polarized and s-polarized light. The methods for calculating  $D$  are well known for lamellar structures.<sup>17</sup> The data are compared to the calculated values of  $D$  for reflection from a 3 nm native oxide/12 nm GaAs/228.5 nm Glass/GaAs structure and, for reference, the calculated values of  $D$  for structures with an 11 nm and 13 nm template layer. Changes in GaAs template layer thickness alter the shape of the spectrum, with the changes in  $D$  being wavelength-dependent. Inspection of Figure 2 reveals that though the shape of the spectrum changes appreciably with a change in the template layer,  $D$  is relatively insensitive to changes in the GaAs template layer thickness at a measurement wavelength of  $1\text{--}520$  nm. The change in the spectrum due to a difference in GaAs template layer can be contrasted with that

due to the presence of a native oxide. The effect of a native oxide covering the surface of the GaAs template layer on the ellipsometric spectrum is given in Figure 3. If the oxide, which has a lower index of refraction than GaAs, is neglected in the calculation the polarization shift is overestimated over the entire wavelength regime investigated. The correct GaAs-on-insulator structure can therefore be arrived at by simul-

Figure 2: Spectrally-resolved ellipsometry measures the GaAs template layer thickness to be 12 nm with a precision of 1 nm. The thickness of the top native oxide is assumed to be 3 nm in the simulations



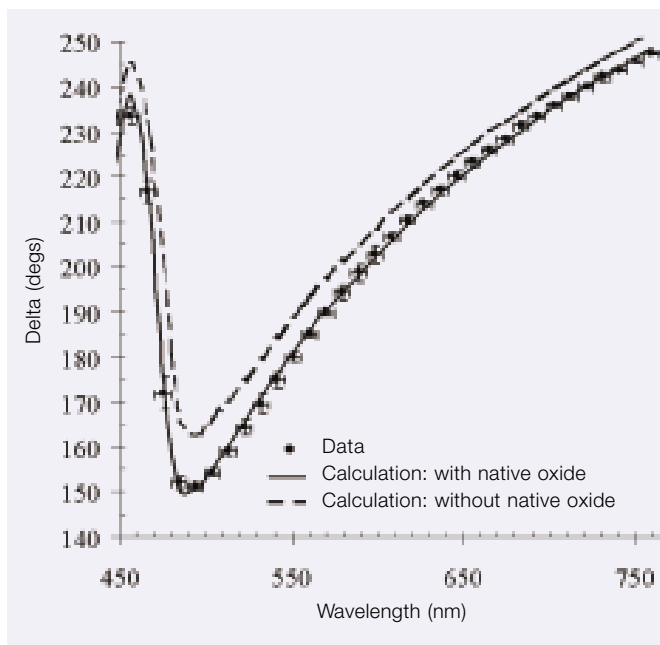


Figure 3: The effect of neglecting the top 3 nm native oxide layer in the ellipsometry calculation is to overestimate the phase shift between polarized components over the entire wavelength regime

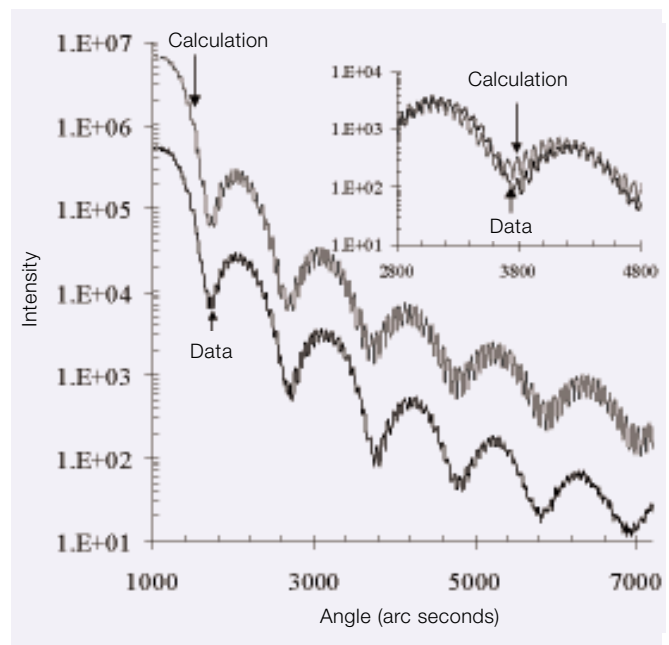


Figure 4: Experimental high-resolution x-ray reflectivity behavior of the ultrathin GaAs-on insulator structure compared to calculation. The calculation has been offset 1 decade for clarity. The non-offset inset shows the level of agreement in the fine fringe structure between the data and calculation. The calculation is for a GaAs-on-insulator structure with the GaAs layer thickness of 11.8 nm and the glass layer thickness of 228.5 nm

on-insulator structure for which the template layer is 11.8 nm, the insulator is 228.5 nm thick, and for which there is a 2 nm native oxide layer. The model calculation was performed assuming ideally smooth interfaces. The effect of including layer roughness in the calculation would be to broaden the periodic features and to slightly change the intensity envelope of the reflectivity profile. The periodicity of the reflectivity oscillation is largely unaffected by the presence or absence of layer roughness. The period of the high frequency oscillations are most sensitive to the glass thickness while the period of the lower frequency modulations are most sensitive to the template layer thickness.

The primary effect of the native oxide layer on the GaAs template layer is to shift in angle the calculated periodic features. The absolute angular position of these features is a function of the average density over the first ~10 nm of the structure seen by the evanescent wavefield during total external reflection at low angles (<1000 arcseconds). The absolute angular position is also very sensitive to slight misalignment of the sample and the resulting "spillover" of the incident beam at low angles. The difference in the thickness of the native oxide derived from either the

ellipsometry (3 nm) or the HRXR data (2 nm) is due to the aggregate uncertainty in the optical constants, density of the oxide, and the influence of slight misalignment of the sample during the x-ray measurements. Further work would be needed to quantify the structural properties of the native oxide present. However, the degree of agreement (neglecting spillover) between experiment and calculation over five orders of magnitude in reflected intensity is evidence that the structure closely resembles the lamellar structure used to model both the HRXR profile and the spectrally-resolved ellipsometry data.

The surface morphology of the GaAs template layer after the removal of the final etch-stop structure by exposure to HF was examined by AFM. Figure 5 shows the web-like morphology that resulted on the etched surface. This morphology persists after additional exposure of the surface to HF. Additional HF etch times of 60 seconds, which is ten times longer than required to remove the final 0.25  $\mu\text{m}$   $\text{Al}_x\text{Ga}_{1-x}\text{As}$  layer, does not alter the surface features. Auger spectroscopy of this surface shows the presence of Ga, As, and O, but no evidence of Al. The persistence of these features even after long exposures to HF suggests that it is not an oxide, but

rather regions of GaAs, excess Ga, or As that are not readily oxidized and removed in the HF solution. It has been demonstrated<sup>18</sup> that exposure of the GaAs surface to HF solutions can result in an As-rich surface rather than a Ga-rich surface. Their previous studies therefore suggest that the material comprising the observed web-like morphology is more likely to be As or GaAs than Ga. The observed surface structure is unsuitable for high-quality epitaxial growth and further chemical processing is needed prior to growth.

Subsequent improvements in the surface morphology must be done by a method that will not consume or impact the 10 nm GaAs template layer in an uncontrolled manner. Native oxides on GaAs surfaces are well known to form slowly.<sup>19</sup> A self-limiting chemical treatment suitable for modifying the 12 nm template layer has been developed<sup>20</sup> that utilizes an exposure of the surface to an aggressive oxidizing environment, followed by exposure to an acid that removes only the oxide. This is a "digital etch" approach to modifying the template in that the two-step process removes a discrete quantity of material (~2 nm) corresponding to the depth of oxidation in the first step of the process.<sup>20,21</sup>



**Before Digital Etch**

$R_{rms}$ : 2nm

**After Digital Etch**

$R_{rms}$ : 0.3nm

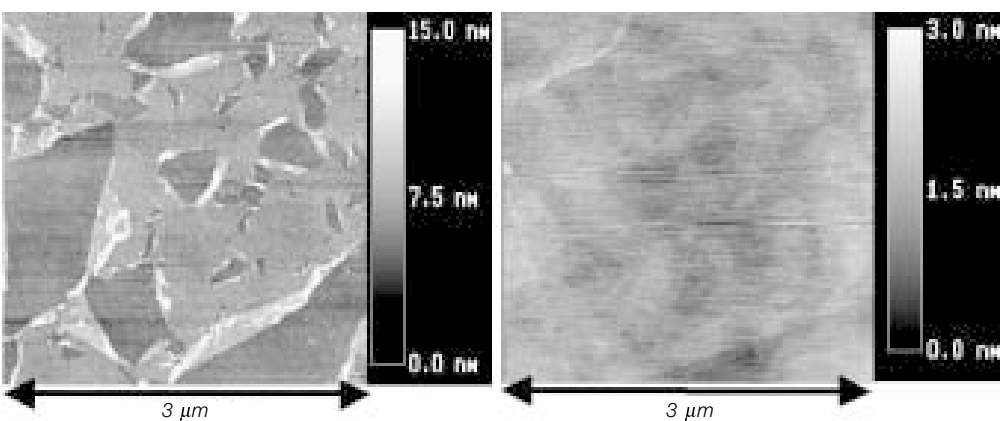


Figure 5: AFM micrographs showing the morphology of the 12nm GaAs template layer. On the left is the morphology present after removing the 0.25  $\mu\text{m}$  Al<sub>0.70</sub>Ga<sub>0.30</sub>As etch stop layer with HF. This morphology persists even after exposure to HF for 10 times as long as is nominally necessary to remove the etch-stop. On the right is the morphology of the template layer after one digital etch cycle. This morphology is not seen to improve after subsequent digital etch cycles

The surface morphology of the digitally-etched GaAs template is shown in Figure 5b. This surface was formed by (1) initially removing the Al<sub>x</sub>Ga<sub>1-x</sub>As etch-stop through exposing the sample to a 1:1 HF: H<sub>2</sub>O solution for 15 seconds, (2) rinsing in flowing DI H<sub>2</sub>O for 30 seconds, (3) exposing sample to a solution of 1:1 30% aqueous H<sub>2</sub>O<sub>2</sub>: H<sub>2</sub>O for 2 minutes, (4) rinsing in flowing DI H<sub>2</sub>O for 30 seconds, (5) exposing the sample to a solution of 1:1 HCL: H<sub>2</sub>O for 2 minutes, and finally (6) rinsing again in flowing DI H<sub>2</sub>O for 30 seconds. The surface web-like structure is not present and a uniform surface morphology results. The r.m.s. roughness of the surface also decreases from 2 nm to 0.3 nm over a 3x3 mm area. This improvement in the surface morphology was realized after one digital etch cycle.

The final surface has a r.m.s. roughness comparable to that measured on commercial, epi-ready GaAs substrates. The efficacy of multiple digital etch cycles was also investigated. The remaining GaAs template layer thickness was measured by ellipsometry between each digital etch cycle for a sample exposed to 4 digital etch cycles. The average amount of material removed in each cycle was measured to be 1.7 nm with a standard deviation of 0.5 nm.

No significant improvement in the quality of the surface as measured by AFM was observed over that obtained by one digital cycle.

In conclusion, it is possible to achieve GaAs-on-insulator structures with a GaAs layer 10 nm thick and with a surface roughness of 0.3 nm. The structures can be characterized by spectrally-resolved ellipsometry, x-ray reflectivity, and atomic force microscopy.

These measurement techniques, combined with quantitative models, allow for the determination of the layer thicknesses and surface roughness of the final structure with high precision and accuracy. The reader who is interested in the impact of such a substrate structure on the relaxation of a subsequently grown heteroepitaxial film is referred to the literature.<sup>22,23</sup>

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# Case study

## Tube Products Oldbury

### Introduction

Tube Products, a division of Tyco Tubing Ltd. are manufacturers of a range of seam welded tubular products for automotive applications, viz exhaust systems and transmission shafts. These products are manufactured from steel strip by a high frequency electrical resistance welding (e.r.w.) process in a variety of widths and thickness, depending on the final tube diameter.

In the resistance welding processes a weld is achieved by a combination of heat and pressure. The required heat is provided by a flow of electrical current through those portions of the workpiece which are to be welded. Consequently in these areas of the workpiece in the immediate vicinity of the weld, the temperature of the material will reach the melting point in order that fusion of the butting surfaces can take place. The chief characteristics of the resistance welding process are:

- 1) High production rate
- 2) No additional metal is necessary for the weld
- 3) Complete control of the welding parameters is possible

As a consequence of 1 and 3 above it is important that those process parameters which have an effect on the integrity of the final weld are carefully monitored. In this respect, metallographic examination of weld cross sections has been in use as the tool to monitor not only the accuracy of the weld mill set up but also to monitor quality during production runs.

### History

Prior to 1998 seam weld cross sections were prepared by "traditional" methods ie. a sample of the formed tube was sent from the weld mill to the laboratory where a weld cross section was cut and prepared for optical microscopy by mechanical grinding and polishing. The primary

objectives of the metallographic examination were:

- 1) assessment of weld integrity
- 2) assessment of butting edge alignment
- 3) check weld dimensions

The entire procedure at that time could take something in the region of 30 minutes, and as a consequence the weld mill could be idle for significant periods of time awaiting results to confirm that the mill set up was satisfactory for starting production runs. In addition, during production runs, the length of time taken for results feedback meant that in the event of a mill condition deteriorating such that there was a drift in weld quality then a considerable volume of lower quality product could be manufactured before remedial action could be taken on the basis of the weld metallography.

### Action

In more recent years, developments in the automobile industry have resulted in an increased demand for high ductility tube which was capable of withstanding severe manipulation. To satisfy these demands required tubing with a highly ductile seam weld and to achieve this it was necessary to keep extremely close control of the weld integrity, butting edge alignment and weld dimensions. Tube Products had always recognised the importance of the relationship between the results from the metallographic examination and tube mill set-up, but with the increased customer demands, the speed of setting up had to be improved to increase mill availability and to avoid serious bottlenecks in the production system.

To improve upon the existing situation Tube Products identified that any replacement system for providing tube weld cross sections had to satisfy the following criteria

- 1) it should be capable of providing rapid results feedback to the weld mill.
- 2) it should be capable of providing consistent results
- 3) it should be simple to operate
- 4) it should be economic
- 5) it should be reliable, robust, and suitable for a shop floor environment

In a radical approach to meeting these requirements, it was proposed that electrolytic polishing equipment could satisfy all these criteria, and after preliminary trials the Struers LectroPol-5 was installed in the exhaust tube manufacturing cell at Tube Products Oldbury.

As stated earlier it was proposed to site the test cell on the shop floor in the immediate vicinity of the weld mill and as such the LectroPol-5 had a number of additional benefits over mechanical preparation methods viz.

- It required a relatively small working spaces. Space on the shop floor was at a premium.
- It only required a 240 volt electrical supply
- No compressed air or water/waste required.

After a short training period for the operators, the speed and ease of operation of the LectroPol-5 has made it possible to provide results from weld cross sections in less than 5 minutes. This significant reduction in section preparation time, has as a consequence, greatly improved mill utilisation and has been an important factor in allowing Tube Products to meet the increased demands from their customers.

As a result of this initial success Tube Products have installed two other LectroPol-5 units and a further two LectroPol-5 units have been purchased for use within the Tyco Tubing operations.

The original pilot set up on the exhaust pipe mill has been improved to

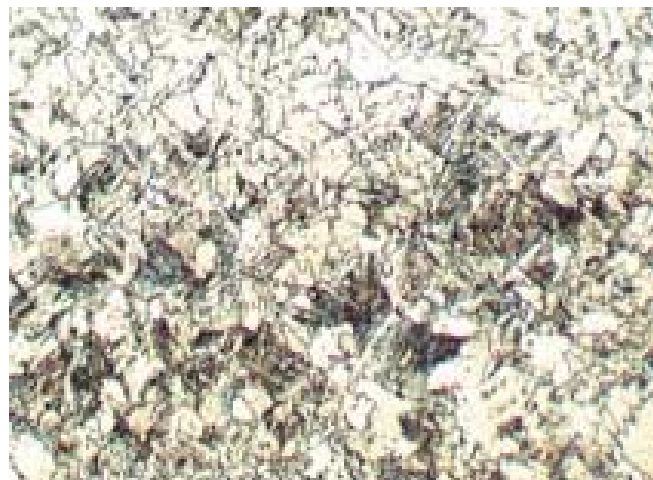
provide a self-contained test station which contains:

- Struers Labotom cut-off machine
- LectroPol-5
- Microscope
- Weld quality comparison/rating charts

The test station is operated by the mill crew, who are now fully trained, not only in the operation of the preparation equipment but also in the interpretation of the seam weld quality by optical microscopy.



*Resistance weld cross section  
50 x*



*Fusion zone  
200 x*



*Resistance weld  
cross section  
50 x*



# New Final Polishing Suspensions, OP-AA and OP-AN

Providing an absolutely scratch-free and deformation-free surface

## Introduction

Polishing with oxide grains is used particularly as the final step of metallographic preparation or used for pre-polishing as a substitute for diamond. The purpose being to completely remove any scratches or deformations produced in preceding preparation steps. With modern high technological materials, the requirements to purity, cleanliness, equal distribution of alloying elements and phases are very important. Thus, a final polishing step with an oxide polishing suspension is often carried out to achieve the necessary surface quality for specimen examination.

## New suspensions

OP-AA and OP-AN are some new final polishing suspensions. They are both based on aluminium oxide abrasives. OP-AA is an acidic aluminium oxide suspension with a pH of 3. It is chemically active and especially suited for the final polishing of ferrous metals, Ni-based alloys and ceramics. The suspension can be mixed with additional acid to increase its chemical activity. OP-AN is a neutral aluminium oxide suspension with a pH of 7. It can be used for all materials. OP-AN is an all-round oxide polishing suspension with the possibility of mixing with additives to obtain chemical activity.

OP-AN can be customized to polish all materials, e.g. with an acidic solution to lower the pH or an alkaline solution to raise the pH.

## Additives

OP-AA and OP-AN can be used with addition of chemicals and reagents to increase the chemical activity of the respective suspensions. For example, an acidic solution e.g. oxalic acid can be used for lowering the pH and an alkaline solution e.g. potassium hydroxide to raise the pH. Additionally, additives can be used for oxidation e.g. hydrogen peroxide, and for lubricating e.g. glycerine.

## Final polishing with oxide polishing suspensions

The oxide polishing suspensions work both mechanically and chemically on the specimen. This means that the oxide suspension reacts chemically with the surface of the specimen, building a reaction layer, which is relatively brittle. This brittle layer is removed by the very small abrasive particles in the suspension. This mechanism is repeated continuously, resulting in a very high material removal, considering the abrasive grain size.

In practice, oxide polishing suspensions leave behind a deformation-free surface.

The oxide polishing suspensions are

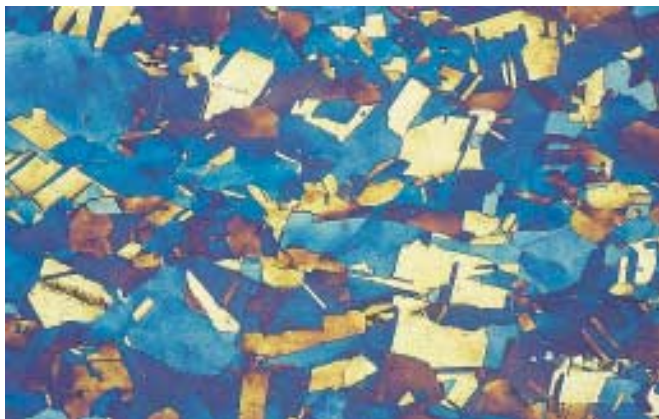
often used on the MD-Chem polishing cloth, to obtain good surface planeness and edge retention. The cloth is resistant to chemical attack in cases where additives are used. MD-Nap can be used as an alternative, where a more gentle final polish is required.

## Additional types of oxide polishing suspensions

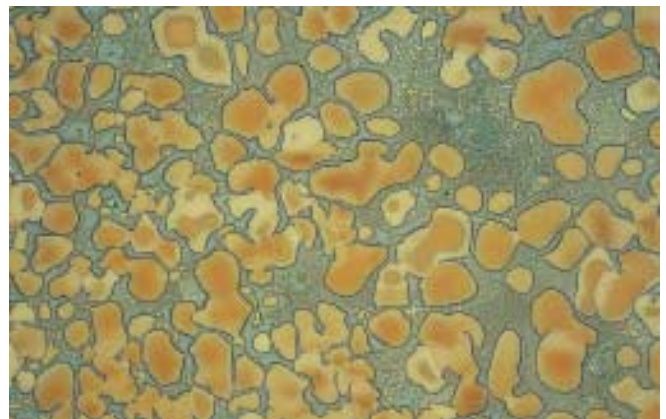
For other purposes and types of materials, additional oxide polishing suspensions can be used. OP-S is a chemically aggressive suspension, like OP-AA, but is based on colloidal silica. OP-S has a pH of 9.8 and is especially suited for polishing of very ductile materials, such as refractory metals, aluminium, titanium but also other materials like monocrystalline silicon. OP-S can also be used with the addition of reagents, normally used for etching the material in question.

OP-U is a colloidal silica suspension with a pH of 9.8, like OP-S, but with a slightly different composition which makes it a milder polishing agent. OP-U is used for heterogeneous non-ferrous materials and composites.

X 10 CrNiNb 18 9, stable austenite, 200x, etched with Beraha II



Cu with 4.5% P, sand cast, 200x, etched with Klemm III



# Duramin Microhardness Tester

## More than just hardness testing

### What is hardness?

Hardness is a simple-to-measure material attribute with which to differentiate between and describe materials and their physical condition during processing. Hardness is defined as the resistance of a material to indentation by a body made of a harder material. Accordingly, most conventional hardness tests involve a hard indenter (e.g. one made of diamond) which is pressed vertically into the surface of the sample. The method most commonly used in test laboratories is the Vickers hardness test. The indenter used in this technique is a square-based diamond pyramid with included face angles of 136°. The advantage of this indenter geometry is that the law of proportional resistance is obeyed. That is, the applied test force is directly proportional to the indentation area, making a Vickers hardness value fundamentally independent of the chosen test force. A minor dependence on the applied force is found for very small indentation depths, but this has other causes. The Vickers hardness value is calculated with the formula  $HV = 0.102 \cdot F/A$ , where F is the applied force and A is the contact surface area of the resultant indentation after the indenter has been withdrawn. The area is computed from the mean diagonal d of the indentation (d = mean average of the two measured diagonals d1 and d2).

*Duramin-20 the fully automatic micro-hardness tester*

### The Duramin series

The new Duramin microhardness testers from Struers represent a new dimension in hardness testing.

### Duramin-1 and -2

are state-of-the-art micro-processor-controlled hardness testers which

enable Vickers and Knoop hardness tests to be performed rapidly, accurately and reliably in both industrial research (metals, sintered materials, ceramic products, integrated circuits, coatings, grain microstructure analyses) and in quality control environments (heat-treated surfaces, cutting tools, wires, small-scale precision-engineered components).

They are particularly suitable for:

- examining the different phases in heterogeneous alloys (e.g. distinguishing between microstructure hardness, polycrystalline hardness and monocrystalline hardness)
- demonstrating diffusion processes
- verifying increasing hardness as the alloy concentration in a mixed crystal is raised



Figure 1: Low magnification image of a weld seam



Figure 2: Detailed image of the weld seam with hardness indentations

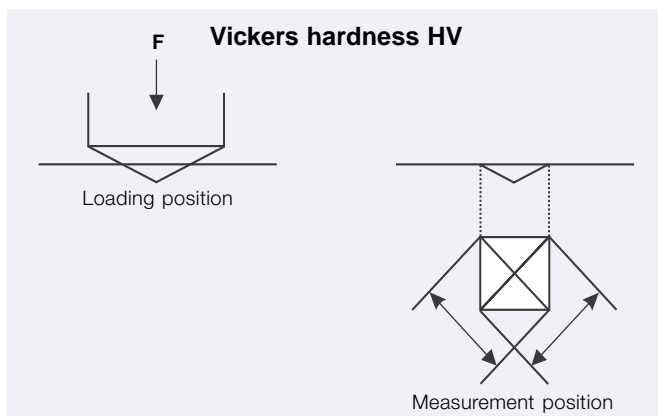


Figure 3: Low magnification image of an induction-hardened component



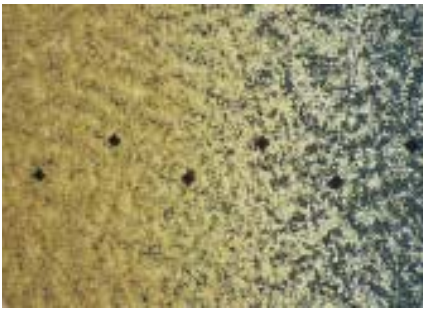


Figure 4: Transition zone with hardness indentations

Figure 8: Hardness indentation on the tip of a needle

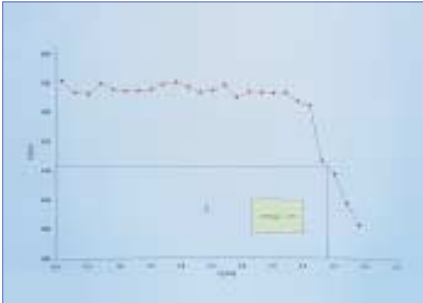
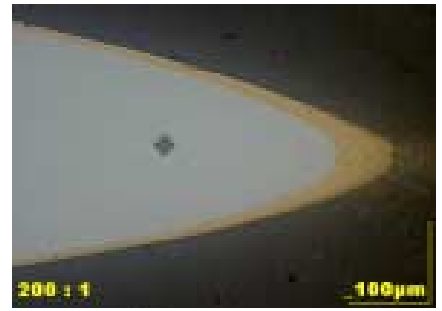


Figure 5: Hardness profile of the induction-hardened component

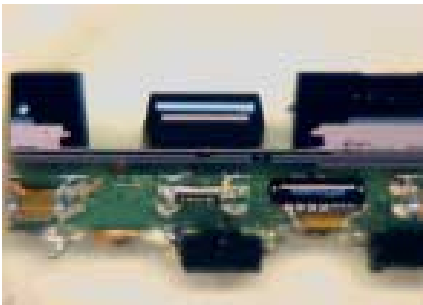


Figure 6: Low magnification image of an electronic component



Figure 7: Detailed image of the component, indentations on the copper strip conductors

- mapping hardness zones from the surface into the body of the sample
- hardness measurements on thin films
- hardness measurements on components with a very small test surface area
- hardness testing of parts whose surface must not be damaged to any large extent.

### Automation of the hardness testing process

Computer-controlled hardness testing systems are now an established part of a modern materials testing

laboratory. The most important reasons for this development are:

- the time savings achievable with large test series and frequently recurring test procedures
- the objective evaluation of measurement parameters (no longer observer-dependent)
- the reduction in operator errors
- the reduction in the amount of physical effort required from laboratory personnel
- the acquisition and storage of measurement data for components for which documentation is obligatory
- the simple production of measurement logs

The number of separate measurements needed in order to establish hardness varies over a large range and depends upon the sample and the definition of the particular measurement problem. Typically, six to ten measurements will be sufficient to determine local case hardness.

For weld seams, however, the number can be many times greater. If conventional hardness testers are used, the tests are carried out manually making the process highly cost- and time-intensive. In addition, the process can be tiring for the test personnel, because despite being a routine operation, the measurements must be performed to a high level of accuracy, so that measurement values are often dependent on the experience of the operator and on the day-to-day variations in operator accuracy.

By employing automated image analysis and by automating the test procedure wherever possible, this source of operator error can be reduced to a minimum. With a modern tester, the operator can concentrate on setting up the sample and then invoke a predefined test sequence. Struers has been actively developing modern hardness testing systems for many years, placing emphasis on ergonomics, user-friendliness and

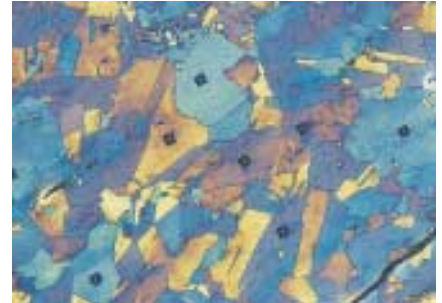


Figure 9: Hardness indentations at low load in individual grains



Figure 10: Indentations in the layer applied at low load. The indentation in the base material is produced using a higher load

the greatest possible degree of automation. These principles also underlay the development and realization of the semi-automatic hardness testing system **Duramin-10** and the fully automatic **Duramin-20**.

The tester is controlled via the serial interface of a computer running special control and image processing software. The system enables the indentations to be made according to a preset series of patterns which are then subsequently measured by the system's image analysis function. The high degree of automation means a significant improvement in the reproducibility of the measurements and an almost total eradication of operator errors.

Typical applications of these hardness testers are:

- measuring case hardness
- hardness profiles along weld seams
- scanning surfaces in order to achieve a topographic representation of surface hardness.



# From Our Lab to Yours



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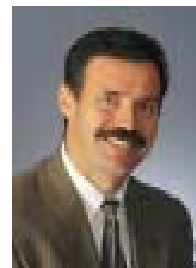
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On the occasion of the 125 years' anniversary of Struers we have been looking through all past issues of Structure to see what kind of tips and advice had been presented throughout the years.



Already in Structure 5, which was printed in 1982, this column was introduced. Back then it was called Ask **Ingeborg Liebl**.

Through her long practical experience in Metallography Ingeborg Liebl could give advice on all different kinds of problems which could arise in connection with sample preparation. In Structure 16, published in 1988, the title was changed to the one still used today, **From our lab to yours**. From this issue on not only Ingeborg Liebl, but also all the other metallographers working in our application laboratories around the world contributed to this column. The following tips and hints are a small selection and they have all been published before, they only have been edited to reflect the changes in the names of equipment and consumables.

## Which diamond cut-off wheel should be selected for cutting composite materials and ceramics?

There are two main types of diamond cut-off wheels: metal bonded and bakelite bonded. Metal bonded wheels, for which the most common bonding metal is bronze, are highly stable and suitable for cutting glass, quartz, crystals, brittle ceramics and minerals and small pieces of sintered carbides. However, for cutting larger cross sec-

Fig. 1





Fig. 2a



Fig. 2b

tions or specimens of composite materials, bakelite bonded diamond cut-off wheels are recommended. Because of its soft bonding medium this wheel will readily cut difficult composite materials and is universally applicable to minerals, all ceramics, glass, sintered carbides, etc. It will generally produce a better surface finish (Fig. 1) than a metal bonded diamond wheel, however, the softer bond shortens the service life.

### How does one clamp delicate materials in without damaging them?

Delicate parts, balls and very irregularly shaped materials often present problems of clamping. The problem is eliminated simply by clamping the material between two plates of expanded polystyrene (Fig. 2). This prevents the material from slipping and breaking during cutting. Compared with, for example, rubber or other plastics materials expanded polystyrene has the further advantage of not settling on the cut-off wheel where it might cover the abrasive grains.

### How to avoid pull-outs from the surface of a specimen during cutting?

This may be avoided by

1. Mounting the specimen, size permitting. In the case of corrosion layers on the surface impregnation with Epofix is preferable.
2. Wrapping adhesive tape around the specimen.

3. Applying preservative varnish or nail varnish on the specimen surface (spray or brush on). It is important that the cut-off wheel should cut from the coated surface into the specimen. Where the entire periphery of the specimen is coated, the latter has to be turned. For this purpose a rotating specimen holder is ideal.

### A simple method for mounting powdered metal

Traditional mounting methods for powdered metals are often ineffective due to settling of the particles. The most common method of mounting is that of mixing the powder directly into epoxy. The resultant mount has only a thin layer of material to prepare. Since most mould cups have slightly curved bottoms, the finished sample ends up looking like a "bulls-eye" with particles only around the outside.

Fig. 3a



Fig. 3b



Fig. 3c



More importantly, if the metallographer must prepare the mount, there is often not enough material layer left. The method that Steve Glancy developed for the MPIF (Metal Powder Industries Federation) is as follows:

- 1) Fill a small plastic vial or any kind of plastic tube with the powder to be examined (Fig. 3a). This method gives a fairly true distribution of particle size (traditional methods are subject to segregation).
- 2) Place the vial into the Epovac. Fill the vials using normal operating procedures (Fig. 3b). This fills all voids in between particles and also within particles.
- 3) Once the epoxy has cured, cut the plastic vial off using a razor knife.
- 4) Mount slug in MultiFast using ProntoPress-10 or use any other mounting resin hot or cold (Fig. 3c). Take care when cold mounting that the slug does not float.
- 5) Prepare mounts.

### Problem of etching steel foils after hot mounting

Etching of mounted specimens with strong acids will often cause problems. Small quantities of undesired polishing medium and abraded material in the spaces between the specimen and the mounting resin can cause reactions which make the etched specimen unsuitable for examination.

The problem can be overcome in the following way:

Alternate the steel foils with PVC foils and place them in a grinding clamp. Heat the clamp with hot water and tighten it immediately in a vice.

This way the steel and PVC foils are pressed so closely together that absolutely no space is left between them. (Fig. 4) Grind and polish in the usual way.

In this connection another problem

Fig. 4

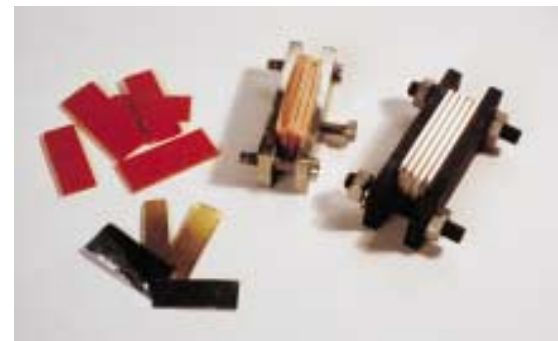




Fig. 5a



Fig. 5b

can be mentioned:

Bleeding of etchant from scratches, pores and cavities, which may cause trouble during drying.

This nuisance is avoided as follows: After polishing, clean the specimen. If the etchant to be used contains water, flush the specimen with distilled water, if it contains alcohol, flush with alcohol, and then immerse it in the solution.

After etching the specimen is rinsed thoroughly and then cleaned with alcohol. When using an ultrasonic cleaner, put the specimen in a small glass beaker with alcohol and place the beaker into the ultrasonic cleaner. After cleaning the specimen is dried with air (do not use hot air) and placed face down on a clean filter or lens tissue.

For macrostructure examination and for examinations up to 400x a protective lacquer (Protecting Lacquer) may be very helpful. Directly after drying the specimen in cold air cover it with protection lacquer and spray into all scratches and cavities. Allow any surplus lacquer to run off and let the specimen dry in a horizontal position.

### Why does Epofix sometimes fail to cure properly?

The surface of the mount tends to be sticky and does not seem hard enough. Always make sure that resin and hardening liquid are mixed very

thoroughly. It is difficult to mix small amounts (e.g. 8 ml resin and 1 ml hardening liquid) sufficiently to obtain proper curing. It is therefore advisable to mix larger quantities, say, 16 and 2 ml. You may further promote hardening by covering the specimen while it is curing, so as to prevent any reaction between the surface of the specimen and the surrounding air.

### How can you prevent specimens from turning or falling over during mounting?

Fixation clips (Code: CLIPS) ensure that thin plates, foils and wires remain upright (Fig. 5a). For irregularly shaped specimens double-adhesive foil (Fig. 5b) can be used to hold the samples upright.

Liquid adhesives may react with some types of cold mounting media. Do not use a fast-curing hobby cement with epoxy resins because they will blister and turn brown during curing.

Samples too small to be fixed by a clip (e.g. some electronic components) may be held in a vertical position during embedding by cementing them to the bottom of the mould with a trace of fast drying cement.

### Protecting Lacquer Solves some Cold Mounting Problems

Cold mounting is very often used for difficult to handle specimens. However, the process of cold mounting can create its own problems, such

as “knocking” the specimens over or “floating” the specimens away from the base.

There are all kinds of devices for holding the specimen in the mount such as “clips”. But while these may help solve some problems they can also create additional problems. As a metallographer I prefer to have only the specimen material in the mount, as this avoids preparation problems and aids examination.

Therefore I prefer to coat the bottom of the mould with a thin layer of Protecting Lacquer and position the specimen in the wet lacquer until it hardens. This technique will firmly hold the specimen on the bottom of the mould in any required position or angle ready for mounting.

The photo shows - from left to right: (Fig.6)

1. Fine wire specimens which should be as upright as possible for cross section examination.
2. Coated electronic components for layer thickness measurement and examination using taper section technique.
3. Thin metal foils which have now been prevented from floating, and are free from risk of damage from clips.

### Useful hints for warm embedding

When warm embedding samples with acrylic resins, i.e. thermoplastic resins, cracks and/or “cotton wool” often occur at the core of the mount (Fig. 7a). This can be avoided by re-

Fig. 6







Fig. 7a



Fig. 7b

ducing the amount of cooling fluid entering the press and prolonging the cooling cycle.

The surface of some warm-embedded bakelite mounts may, on contact with alcohol, become delicate and brittle. The reverse side may also assume a pale colour (Fig. 7b). This defect usually occurs during the mounting process when the heating time is too short or the temperature too low.

Specimens should be degreased prior to mounting for good adhesion between the embedding medium and the specimen. This is easily achieved with alcohol. Degreased specimens should only be handled with a pair of tweezers to avoid any transfer of finger perspiration to the material.

### Tips on Hot Mounting

Getting it right at the hot mounting stage provides the opportunity to improve sample preparation further. Therefore, the following tips have been provided to help all metallographers improve their mounting techniques and, ultimately, their preparation results.

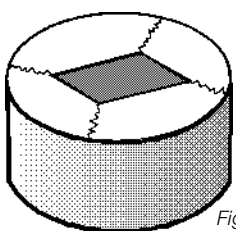


Fig. 8

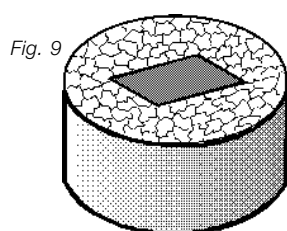


Fig. 9

### Individual grains of resin still visible after mounting

This usually occurs when pre-heating for a long period of time with little or no pressure has been

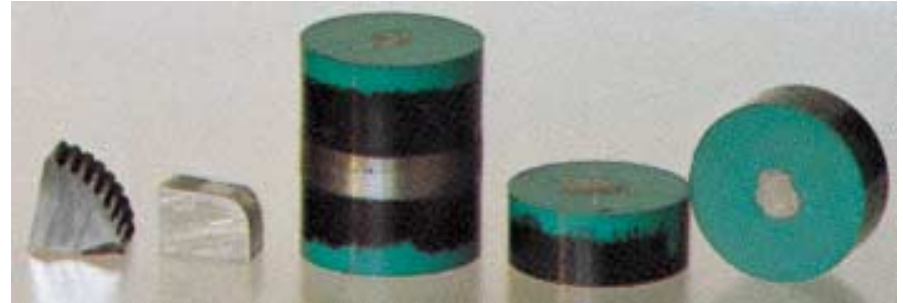


Fig. 10

### Staining and scratching after polishing

It is important to degrease all samples in alcohol and dry thoroughly before mounting, as trapped dirt and grease result in adhesion problems, and finally gaps between the sample and the resin. During preparation these gaps hold quantities of suspension, lubricant and dirt which affect the final polish and any subsequent etching by staining and scratching the surface.

### Radial cracking

Sharp edges or corners set up stress concentrations in the resin which upon cooling produce cracks that cause similar problems to gaps. Grinding sharp edges/corners prior to mounting reduces the stress concentration and, therefore, eliminates cracking (Fig. 8).

### Shrinkage within rings

Mounting ring shaped samples usually results in a higher shrinkage inside the ring than outside. If possible, it is always better to cut the ring first in half and then mount each half slightly apart from each other. This will significantly decrease the shrinkage experienced at the inner surface of the ring.

carried out using a thermosetting resin. It is always best not to preheat thermosetting resins, as they cure upon heating. If pre-heating is required, a thermoplastic resin should be chosen (Fig. 9).

### Mounting fragile samples

To avoid destroying fragile samples, a thermoplastic resin should be used incorporating a pre-heating step of about six minutes with no pressure. This should be followed by a short heating time and long cooling period, both with low pressure. Pre-heating with no pressure will allow the resin to melt, so as when pressure is applied during the heating cycle it will be transferred to the samples isostatically therefore, considerably reducing the chances of destroying the samples. The long cooling period will ensure that any internal stresses are relieved completely.

### Mounting porous specimens

Porous samples should be mounted in the same manner as fragile samples, however, with porous samples it is advantageous to place a small amount of resin on the lower ram before placing the sample. During the process this resin on the lower ram will melt and be pushed into the pores.

### Is it possible to reduce the process time of hot mounting?

No, but in case of compact routine specimens use of an intermediate ram permits simultaneous mounting



Fig. 11

of two specimens (Fig. 10). However, the height of the specimens must not exceed 10 mm.

AntiStick stearate powder should be applied. Compressive force and time need not be increased very much.

### Colour Coding of the Mount

The MultiFast range of resins are available in three different colours: green, brown and black. This consequently provides any lab with the opportunity to ease identification problems by colour coding their samples with a high quality, general purpose resin at a low cost (Fig. 11). The Struers Application Guide for Hot Mounting gives more hints and tips on both the hot mounting process and specific resins. This handy booklet can be ordered from your local Struers dealer.

### How does one make sure that very porous specimens are not damaged in the preparation, and which is the most suitable method of drying such specimens?

Specimens with cavities, cracks and pores may advantageously be mounted in Epofix resin in the vacuum impregnation apparatus. An advantage of this resin is its very low hardening shrinkage.

During the preparation care should be taken not to remove too much material, in case the impregnation layer is also removed.

Ultrasonic cleaning may be recommendable. After cleaning the specimen should be rinsed in pure alcohol, dried only briefly in a cold

Fig. 12a



flow of air and sprayed with Protecting Lacquer

### How does one hold irregular specimens for automatic grinding and polishing?

1. In the case of specimens of heights between min. 5 mm and approx. 15 mm our double-adhesive tape (Code: MATAB) can be used for fastening the specimens onto the plain specimen holders. Where several specimens are to be prepared simultaneously, they should be of the same height and be evenly distributed over the area of the specimen holder disc (Fig. 12a).

The specimens may be cut to a uniform height on the Discotom cut-off machine by clamping the material to be cut in only one of the two vices, clamping a stop block in the other.

2. The mounting of specimens thinner than 5 mm is best achieved with Lakeside Cement.

This thermoplastic adhesive becomes fluid at 140°C and dissolves completely in acetone, which also makes it suitable for double-sided polishing.

In both cases the specimens are fastened directly onto the specimen holder plate, which has first been heated to 140°C (fig. 12b).

### Manual preparation of thin specimens

Holding a flat, thin specimen by the hand during preparation is practically impossible. Many metallographers solve this problem by gluing the specimen to a dummy

Fig. 12b



Fig. 13



with wax or double adhesive tape. We have discovered an alternative solution which is quick and handy: we use a suction disc of the type which is known from bathrooms and kitchens for attaching towel hooks or soap holders to a smooth wall (Fig. 13). In order that the suction disc may be easily removed after use, there should be plenty of water between the suction disc and the specimen surface. When the disc is moved to the edge of the specimen, the vacuum disappears and the specimen can be removed.

### Useful hints for fine grinding

The finer the abrasive, the thinner the supporting paper. Always store used, wet grinding papers in water, as drying out may cause them to twist out of shape.

Fine grinding paper may be torn by sharp specimen edges during manual grinding. This can be avoided, where the Knuth-Rotor principle is used, by placing a used grinding paper under the new one.

### Preparation of small tubes, balls and other small articles with a diameter of less than 1 mm - exactly in the middle

In order to be able to prepare a specimen - e.g. a small tube, needle, ball etc - exactly at its centre, it is essential that the specimen be planely embedded. The simplest choice is hot compression mounting, because this method provides a plane support for the specimen. Preheating without pressure - by means of Predopress or Prontopress with a timer kit - makes it possible to mount delicate specimens with no deformation.

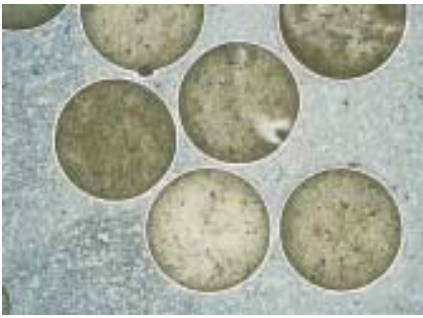


Fig. 14a  
Metal covered synthetic balls.  
Diameter approx. 0.8 mm



Fig. 14b  
Small tubes, longitudinal section,  
material 1.4571, (X 6 CrNiMoTi 17 12 2).  
Laser welded point.  
Dimensions 1 x 0.25 mm. Etching V<sub>2</sub>A-Beize

For cold mounting with Epofix a glass plate, coated with heat-conducting paste, is used as support. Press only the ring of a MultiForm firmly onto the plate, put the degreased specimen into the MultiForm and fill up with Epofix. The mounted specimen must not be ground with SiC-paper. Instead, it should be pre-polished directly on a DP-Plan cloth, using 15 µm Diamond Spray or Suspension. This way, a defined material removal can be obtained, even when preparing very small specimens.

Fig. 14a: Metal covered synthetic balls. Diameter approx. 0.8 mm

Fig. 14b: Small tubes, longitudinal section, material 1.4571 (X 6 CrNiMoTi 17 12 2). Laser welded point. Dimensions: 1 x 0.25 mm. Etching: V<sub>2</sub>A-Beize

### A simple and accurate way to measure the depth of thin layers

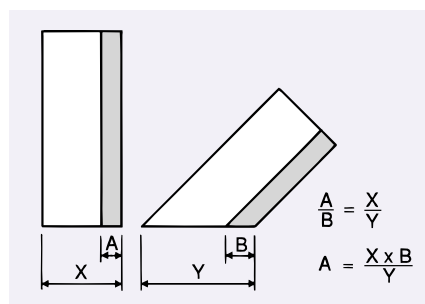
Accurate measurement of very thin layers is often difficult to obtain with conventional measuring instruments. An easy solution to this difficulty can be achieved by using metallographic cross-sectioning techniques for preparing the specimens at oblique angles. Preparing the specimens at these oblique angles exaggerate the layers such that they can be easily measured. This technique is known as taper sectioning. Cutting, or mounting and grind-

ing the taper section will largely depend on the size of the specimen. In the case of electronic components it is best to mount the specimens first at an oblique angle, and then grind the surface away until the desired area for measurement has been reached.

Calculating the actual thickness. Prior to mounting, the total thickness of the specimen (X) is measured using conventional instruments, e.g. a micrometer. Then the specimen is mounted at an arbitrary angle, and is subsequently ground and polished to the desired standard for microscopical assessment and measurement. The total thickness of this prepared taper section of the specimen is now measured (Y). The ratio between X and Y gives us the magnification factor created by mounting the specimen at an oblique angle.

An alternative to using conventional measuring instruments would be to mount two identical specimens in the same mount, one at an arbitrary angle as above and the other vertically, to be used as a reference. In this way both specimens can be measured microscopically with greater accuracy, thus producing a more accurate magnification factor. Now, the layer thickness on the taper section specimen is measured (B). The three measurements X, Y and B can now be used to calculate the actual thickness of the layer (A). The calculation can be expressed as

Fig. 15a



the equation:  $A = X \times B / Y$  where X is the total thickness of the specimen; Y is the total thickness of the tapered and prepared surface; B is the thickness of the tapered and prepared layer; A is the actual layer thickness (Fig. 15a).

Electronic component for layer measurement showing one reference specimen and one taper section glued to the base of mould - prior to mounting (Fig. 15b).

### Why are different lubricants used for diamond polishing?

Alcohol-containing lubricants, such as Blue Lubricant, are always used where fast and effective abrasion is required. Owing to the low content of grease, the diamonds are well cleaned of abraded material during polishing.

For this reason Blue Lubricant is used in the following processes:

1. Fine grinding on MD-Allegro.
2. Polishing of hard and tough materials, such as steel, cast iron, stainless steel, titanium, chromium, nickel, tantalum, sintered carbides, silicon, etc., regardless of diamond grain size and type of cloth.
3. Pre-polishing of relatively soft materials, such as copper, brass, aluminium, zinc, silver, etc. with 6 µm and 3 µm diamond on DP-Dur and DP-Mol polishing cloths.

Being an oil emulsion, Red Lubricant has a greater lubricating effect, thus reducing the abrasive rate of the diamond grains and, consequently, also the depth of scratches. Red Lubricant is suitable for:

1. Final polishing of soft materials on DP-Nap polishing cloths with 1 µm and 1/4 µm diamond paste.
2. Polishing of delicate and difficult materials on all polishing cloths. Red Lubricant should be removed with alcohol. The use of water may cause the formation of a film which is difficult to eliminate.

Fig. 15b





## Cleaning of materialographic specimens

Cleaning of specimens is a frequently overlooked area in materialographic preparation. It is, however, an area which is well worth a closer look as even the best preparation can be ruined by defective or insufficient cleaning. Insufficient cleaning of specimens can lead to contamination, resulting in scratches. Moreover, the microscopic evaluation of an insufficiently cleaned specimen is a virtual impossibility. The following is a description of the tools which should be close at hand during the cleaning process, their applications and the problems which require particular attention. During the cleaning process, the following should be available in the laboratory:

- " Hot and cold water
- " Liquid cleaning agent
- " Ethanol
- " Compressed air
- " A hair drier
- " An ultrasonic cleaner
- " Absorbent cottonwool
- " A soft washing-up brush

### Hot and cold water

Hot water is far superior to cold water for loosening dirt and removing soap. It should be remembered; however, that hot water has a tendency to promote corrosion of the specimen. It is therefore a good idea to use lukewarm water unless it is very difficult to clean the specimens.

### Liquid cleaning agent

Normally it is not necessary to clean the specimens between the various stages of grinding. Water is part of the grinding process and loose grains will be immediately washed away.

The specimen should, however, be rinsed after the final grinding stage in order to avoid transferring abrasive particles to the polishing cloths. When fine grinding with MD-Allegro and when polishing, the specimens must be cleaned in between each step. A neutral, liquid cleaning agent is a considerable help here. Use one drop for each specimen.

### Ethanol

When the cleaned specimens are to be dried, the easiest thing is to rinse their surfaces first with ethanol. Ethanol containing a denaturation agent is suitable for this purpose,



providing that the liquid does not leave a film on the specimen. If it is unavoidable, or if ethanol is difficult to acquire, isopropyl alcohol can be used instead. A plastic spray bottle is a good idea when rinsing specimens with ethanol.

### Compressed air

If the laboratory is equipped with compressed-air, a compressed-air gun can be fitted beside the sink. The compressed-air gun is used to dry the specimens after cleaning in between the various preparation stages. This method is highly effective but cannot, unfortunately, be used for drying after the last polishing stage as compressed air always contains some small drops of oil which are often deposited on the surface of the specimen and appear as small blue spots under the microscope.

### Hair drier

A hair drier is the best option for drying specimens after the last polishing step. In order to ensure that the ethanol is blown away and evaporates quickly, the hair drier should have a powerful fan and an efficient heating coil. The best method is to hold the specimens at an angle and direct the jet of air from the top down. This ensures that the ethanol disappears without leaving stains.

### Ultrasonic cleaner

If the specimen has been mounted, there may be cavities between the specimen and the mounting resin. The specimen may also be quite porous.

In both these cases, abrasive particles can easily collect in the cavities and fall out again during polishing. It is therefore important to remove them before proceeding to the next preparation step. The best solution is a high-frequency ultrasonic cleaner - preferably 50,000 Hz. An ultrasonic cleaner is not necessary if

there are no cavities or pores. An ultrasonic cleaner cannot be used with highly porous ceramics. It will not only remove the loose grains but also damage the structure of the material.

### Absorbent cottonwool

Absorbent cottonwool can be used during cleaning of the specimen with soap.

It is important that the cottonwool is wet in order to avoid the risk of scratching the surface from hard fibres in the cottonwool. A clean thumb may also be used.

### Soft washing-up brush

After fine grinding with MD-Allegro, it can be difficult to get the specimens entirely clean. An easy solution to this problem is to use a soft washing-up brush which can easily get into the corners. The brush may well give rise to some small scratches but these are smaller than those resulting from fine grinding and should cause no problems. The brush must not, of course, be used during polishing.

In a nutshell, the cleaning process is as follows:

1. Rinse the specimens first in lukewarm water.
  2. Apply a drop of a neutral, liquid cleaning agent to each specimen.
  3. Rub the cleaning agent into the surface of the specimen with a swab of wet cottonwool or with the thumb (if using MD-Allegro, a soft brush may be used).
  4. Rinse the specimen under running water. As far as possible, avoid touching the surface.
  5. Rinse the specimen in ethanol.
  6. Dry the specimen using compressed air. Use a hair drier after the final polishing stage.
- Tip: If specimens must not come in contact with water, ethanol may be used instead of water and soap.

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