

Material Analysis

Microhardness

Q Material Testing

#### «editorial»

## Dear Reader,

The FiGB team have had a glimpse of ongoing developments at Helmut Fischer GmbH in Sindelfingen, Germany, having just returned from a recent trip there. And that's not just product development across the broad range of products. Helmut Fischer GmbH has just acquired a building which, when redeveloped, will increase their working area substantially. This, coupled with increasing staff levels across all departments, will help meet the rate of product development as well as increasing production capabilities to keep up with global demand.

There is a constant drive to improve the accuracy and precision of our instruments. The recently launched family of XAN® X-Ray instruments offers an impressive step up in performance from its predecessors. The new range of PCB X-Ray instruments harnesses the expertise and experience that Fischer has gathered over the years into these specialist systems to suit this industry. Part of the range has the capability of measuring a few nanometres of gold coatings across structures that are microns wide.

The handheld instruments have been updated and the range of probes that we can supply keep increasing with more specialised applications. Not only that, but the existing range of probes continue to be developed with features like harder probe tips.

We have also increased our global presence with new offices in South Korea, Mexico and Brazil. These offices are not just sales/service outlets but come complete with Application Labs so that we can better support our customers locally.

All this activity is necessary for the Fischer Group to continue to support its broad customer base and its ever evolving needs.

We hope you enjoy reading our latest issue of Fischerscope<sup>®</sup>...

#### The team at Fischer GB

«closer examination»

## **UK Seminars and Events** Planned for 2013

Fischer GB are keen to meet as many existing and potential new customers as possible and are planning a comprehensive schedule of events, exhibitions and seminars to showcase many of the developments and new instruments from Germany, that are now directly available from the UK. Please refer to the back cover for more details on events.

## What's New for 2013?

#### X-RAY XAN® 220/250

Those familiar with Fischer's XRF technology will be know of the XAN® range. The 220/250 is the result of significant R&D and the silicon drift detector (SDD) is a major enhancement, opening up many new and extensive applications in the field of precious metal and pollutant analysis. Fischer will be demonstrating this instrument at their XRF Seminar on May 1st (see back page for more details)



#### FMP RANGE OF COATING THICKNESS GAUGES

This popular and versatile family of coating thickness gauges have all been upgraded with colour screens as standard.

#### **POROSCOPE®**

The Poroscope is becoming recognised in the corrosion and marine industry as an invaluable crack/pore detector, otherwise invisible to the eye. If this is your area of responsibility, visit us at Pipeline Protection in October (see back page for more details)

#### PCB RANGE OF X-RAYS

This is an exciting new development planned for late spring/early summer. If this is your field, email 'PCB specific X-ray' as the subject to info@fischergb.co.uk



# Tailor-made probes for every application – example SAM application (sprayable acoustic material)



Figure 1: FA14 probe being used to measure SAM coatings (Daimler AG, Mercedes-Benz Bremen factory).

FISCHER offers a very wide range of different types of probe for measuring coating thickness, because it is our stated philosophy to be able to offer a tailor-made solution for as many applications as possible across the widest range of industries.

When we learn of new applications for measuring coating thickness for which there is still no suitable technical solution, we try to develop suitable probes for these applications as well.

A typical example is the quality control process for SAM (sprayable acoustic material) coatings. In the automobile industry, a great deal of effort is taken to reduce the weight of components, while maximising automated pro-duction. In this context, the typical large soundproofing mats in vehicle interiors are replaced by locally and selectively applied SAM coatings. These locally sprayed layers can be easily seen in the passenger footwell (Figure 1). These must be applied within predefined thickness tolerances in order to maintain the targets for weight, build freedom and cost. Typical coatings are between approx. 2 mm and 4.5 mm. These sound-proofing layers are applied as base material to both the steel and aluminium elements. Furthermore, it must be possible to measure the coating thickness even in places that are difficult to access, which requires a compact probe design and as small an edge influence as possible when measuring.

No suitable probe was available for the stated coating thickness range. Probes that were sufficiently compact had too small a measuring range, whereas probes that had sufficient measuring range were too large. Therefore, the FA14 probe was developed as a solution for this and similar applications. This probe works according to the eddy current method (DIN EN ISO 2360), but is designed in such a way that it can be used to measure non-conductive coatings as well as conductive, non-magnetisable (e.g. aluminium) and magnetisable (e.g. steel) base materials. The measuring range is assured on both base materials up to at least 5 mm. The probe was implemented with an angular design (see Figure 2, height 31 mm) so it is also possible to take measurements in hard-to-reach places. The sensor has an external casing diameter of 20 mm. This means that the field focus is optimised in such a way that it is possible to take measurements on a circular section 20 mm in diameter without edge influence or, in other words, as long as the section to be measured is not significantly smaller than the probe itself, it is possible to take measurements without edge influence. The base material thickness plays no role in most practical applications, because it is already possible to measure base material thicknesses, e.g. of aluminium, larger than about 0.05 mm without any influence. It is common knowledge that FISCHER eddy current probes compensate for conductivity, and the FA14 probe is no different. This means that the conductivity of the non-magnetisable base material used plays no role in practice and therefore, for example, different aluminium alloys do not influence the measurement of the coating thickness. To summarise, these are important characteristics which allow for the best possible use of methods such as the SAM application in automotive engineering. In Mercedes-Benz's Bremen factory (Daimler AG), the measurement accuracy of the FA14 probe was successfully proved for use on aluminium as well as on steel.



Figure 2: FA14 probe (operable with all hand-held FMP series instruments).

Our thanks go to Mr Dellwisch in Mercedes-Benz's Bremen factory (Daimler AG), paint process technology KF-OC2 PTL department, who has supported us in the development of this probe by giving us many suggestions and practical measurements.

#### Dr Hans-Peter Vollmar

Measuring point	Target min. [mm]	Target max. [mm]	Actual [mm]
MP1	2.7	3.5	2.8
MP2	2.7	3.5	2.9
MP3	2.7	3.5	3.1
MP4	1.7	2.5	2.0
MP5	2.7	3.5	2.9
MP6	2.7	3.5	2.9
MP7	3.7	4.5	3.4
MP8	1.7	2.5	2.1

Table 1: Example of typical measurement values from quality control of SAM coatings on eight predefined measuring points, in which MP7 lies outside of tolerance (Daimler AG, Mercedes-Benz Bremen factory).



## Instrumented indentation test on small structures complying with DIN EN ISO 14577 using the new PICODENTOR<sup>®</sup> HM500



Figure 1a: New PICODENTOR® HM500: Complete system with active anti-vibration table and measurement unit.

In the area of instrumented indentation testing complying with DIN EN ISO 14577, the trend in many areas is towards measuring ever smaller structures, ever more complex geometries, and ever thinner coatings. In order to be able to solve these tasks in a useful way, it is not just the demands on the measuring instrument that become greater, but the positioning of the probe and the support structure must also become ever more precise and stable. The new PICODENTOR® HM500 is made of granite (Figure 1a) and has the ideal requirements for this, as shown in the following example. Cross-grinding measurement is a task that recurs over and over again. This is to be demonstrated on an approx. 15 µm thick nickel coating on brass. In order to avoid the influence of the surrounding materials (investment material and brass), the distance between the indentation and the edge of the coating must be at least 2 - 3 times the diagonals of the indentation. This represents an equal challenge for the instrument in several respects.

The specimen for cross-grinding is fixed in the HM's grinding specimen holder (Figure 1b). This guarantees that the indentor is in a



Figure 1b: Grinding specimen holder to hold specimens for cross-grinding rigidly.

Figure 2: Impressions on the crossgrinding of a nickel coating approx. 15 μm in thickness (25 mN, 60s, micron scale).

2a: Good positioning, impressions in the centre and in the cross-thread (Martens hardness of the impression in the cross-thread HM: 3,893 N/mm<sup>2</sup>).





### 2b: Bad positioning at the periphery (HM: 2,578 N/mm<sup>2</sup>).

perpendicular position even when specimens are not perfectly plane-parallel and ensures that the object you are measuring does not move. Small forces are needed to obtain sufficiently small impressions. The PICODENTOR® HM500 is ideally suited to this. For tremor-free measurements, the measuring instrument must be protected from vibrations. This is achieved through the stable granite base combined with an active anti-vibration table and a closed measurement unit. In order to obtain an exact position on very small surfaces, the X/Y table must be capable of very high precision and the optics must provide a clear picture. The first requirement is fulfilled by the new HMT5 X/Y table. It has a unidirectional restart accuracy of less than 0.5  $\mu$ m. The improved optics provide a sharper picture of the specimen's surface. As a result, smaller structures can be eliminated. The motorised Z-axis and the autofocus function also make measurement preparations easier.

Figure 2 shows impressions in the nickel coating. In Figure 2a, the programmed position was obtained and the test force was set sufficiently low. The impressions are in a row in the centre of the coating and are sufficiently far away from its edges. The position of the cross-thread demonstrates the accuracy of positioning. It was easier to find the location of very small or less clear impressions (e.g. on very hard materials). Figure 2b shows the results in the case of poor positioning accuracy. The impressions are not central and therefore the other coatings influence the results.

Figure 3a shows the indentation curves for the impressions shown (red: high positioning accuracy, blue: poor positioning accuracy). The markedly higher deviations between the individual measurements can be seen in the case of poor positioning. This is why more measurements are needed and the total measurement time considerably increases. Figure 3b shows the mean values for the resulting Martens hardness: the poor positioning shows up even more clearly in the variation of the Martens hardness results.







3a: Indentation curves in good (red) and bad (blue) positioning.

3b: Resulting Martens hardness. Even when the mean values agree within the distribution, more measurements must be carried out in the case of a bad positioning in order to achieve an acceptable level of accuracy.

#### Summary

Table 1 summarises the measurements and shows mean value andstandard deviation.

Conclusion: A precise positioning, good optics and a motorised Z-axis do not just make measurement more convenient, but also allow for considerable time savings and make the results more solid.

Dr Tanja Haas

	Number of measurements	Mean value N/mm²	Standard deviation N/mm <sup>2</sup>	Coefficient of variation %	Minimum N/mm²	Maximum N/mm²
good positioning	12	3836	127	3.3	3674	4165
bad positioning	15	3,401	911	26.8	281	4,192

### «information from practice»

## Quality control in the manufacturing process with the FDD<sup>®</sup> manufacturing diagnosis diagram

Production control for quality assurance plays a decisive role in all industry sectors, as it does in coating processes. FDD acts as a graphic representation of production metrics, e.g. mean values of coating thicknesses together with their associated distribution in order of priority. It enables discrepancies to be recognised immediately within an overall system. It gives the practitioner a fast and complete graphic overview of production size distribution. FDD is part of the FISCHER Data Center software and of many Fischer measurement instruments.

FDD is the easiest way to monitor and optimise manufacturing processes using «straight line rankings». Any practitioner – even without knowledge of statistical quality management – should easily be able to use this tool to recognise interference factors and avoidable causes of process variations quickly and to eliminate them if necessary. It is also possible to notice at a single glance whether the production process you are observing meets potential tolerance specifications or not.

#### Examples of fields of application for FDD:

- FDD is a simple and effective software tool for adjusting and managing production processes.
- Measurements of parts from different manufacturers, suppliers or batches can be directly compared as part of the incoming goods inspection.
- In research and development, FDD helps to evaluate experimental data and to test possible differences for their significance.

Depending on the measurement strategy selected, FDD can be used to answer a whole range of different questions.

### Example 1: The evenness of coating on a vehicle bonnet needs to be checked.

It is a required quality feature to have the same coating thickness over the entire surface of a bonnet. Measurements are carried out on this bonnet using a tactile probe. An important feature of the FDD rankings diagram is the quality control of a multi-part product. Here «multi-part» means that the bonnet is divided into six measurement areas and each measurement area is regarded as an individual part (see Figure 1).

Three individual measurements are taken in each measurement area. The mean value and the distribution are calculated from the measurements taken in each measurement area. In FDD the mean values are linearised and shown in ascending order on a straight line (see Figure 4).



*Figure 1: The measurement areas with the measurement points on the bonnet.* 



#### **FDD® information content**



#### Four times as bad:

- 1. Tolerance falls short, one point (mean value) is below the lower threshold value (UGW)
- 2. Sharper increase: mean values differ strongly
- 3. Grouping: different point colours, point frequencies
- 4. Large scatter bar (red vertical lines): although almost all mean values lie within the tolerance limits, a considerable proportion of the individual measurements are outside of the tolerance limits

#### The production process can be optimised.

*Figure 2: Simplified representation of FDD.* 



Figure 3: Distribution of the mean values of coating thickness on the bonnet by area. The "green group" (measurement areas 2, 3, 4, 6) differs systematically from the "blue group" (measurement areas 1, 5).

There are no systematic differences between the mean values of the coating thicknesses in measurement areas 2, 3, 4 and 6, and these can be identified in FDD as uniformly coloured (green) points. On the other hand, measurement areas 1 and 5 (blue points) comprise a second group. There are no systematic differences between the mean values of the coating thicknesses in this group either. If FDD shows different coloured points or vertical bars, it indicates that there are systematic differences between the mean values of the coating thicknesses. In the current example (Figure 4), the "green group" can be systematically distinguished from the "blue group". In order to fulfil the tolerance requirements, the user must carry out two interventions. In measurement area 5, the scatter bar is above the upper limit, so the distribution must be reduced here. The mean values of measurement areas 1 and 5 must be reduced. By using the proposed optimisations, the target is for all mean values of the coating thicknesses to be contained in one single group. In Figure 4, this target is represented by a grey vertical bar and can be achieved with the help of FDD's quality diagnosis. The two distribution functions, "green group" and "blue group", are transferred to a wider distribution function, "grey group", by means of suitable manufacturing measures.

## Example 2: Batch consistency of the process for coating the bonnet needs to be tested.

Measurements are taken on eight bonnets in a production run. For this purpose, a measurement is carried out at six measurement



#### Four times as good:

- 1. All points (mean values) lie within the predefined tolerance limits
- 2. Lower increase: mean values only differ slightly
- 3. Uniform distance between the points, no grouping only identifiable by the colour of the points
- 4. Small scatter bar (red vertical lines): all individual measurements lie within the predefined tolerance limits

The production process is optimised.

points (see Figure 1). These measurements are combined into a block, and the mean value and the distribution are calculated. The mean values are sorted into ascending order (by ranking) and projected onto a straight line in the diagram according to their size (see Figure 5).



The points in Figure 5 only have one colour, i.e. there are no systematic differences between the measurements on the different bonnets. All mean values are indeed within the predetermined tolerance, but the scatter bars are above the tolerance limits. This means rejects are to be expected in the production process. The distribution within a single bonnet must also be reduced in order to have complete control of the process (see Example 1).

Mr Rainer Hammermuller



# The silicon drift detector (SDD): instruments and applications

The improved physical characteristics obtained by using the silicon drift detector (SDD) open up new areas of application (see also the article on the silicon drift detector (SDD) in FISCHERSCOPE No. 2/10). These are particularly evident when measuring with very high counting rates, when determining very thin coatings and trace elements, and when analysing elements with low atomic numbers. In the meantime, SDD has therefore found its way into a whole range of applications. Three examples are examined more closely below.

#### Analysis of precious metals

If precious metals are to be analysed with the highest possible sample throughput, as, for example, in testing laboratories or with alloy manufacturers, then instruments with SDD are particularly suitable. Here it is possible to work with very high counting rates and thereby



FISCHERSCOPE® X-RAY XAN® 150

achieve very good repeatability. For practical application, typical gold alloys used in jewellery give mean values of approx. 0.3‰. Therefore an improvement is achieved by a factor of 2 – 3 compared to instruments using PIN diodes as detectors.

Alloy	XDV®-SDD	XAN®150	
Au999.9	0.23	0.22	
Au900Ag50Cu50	0.37	0.34	
Au750Ag50Cu200	0.21	0.36	
Au750Pd100Cu80Ni	0.36	0.50	
Au585Ag45Cu370	0.37	0.38	
Au333Ag75Cu470Zn	0.26	0.37	

 Table 1: Repeatability (60s measuring time) in ‰ for different gold alloys

 measured with FISCHERSCOPE® X-RAY XDVR-SDD and XAN® 150.

#### **Determining pollutants**

Determining pollutants is very important for the manufacturers and distributors of many products. Maximum values, in particular for the presence of heavy metals, are specified in various regulations such as RoHS (Restriction of Hazardous Substances), DIN EN 71 (Safety of Toys) or CPSIA (Consumer Product Safety Improvement Act). FISCHERSCOPE® X-RAY



FISCHERSCOPE® X-RAY XDV®-SDD

XDV<sup>®</sup>-SDD and XAN<sup>®</sup> 150 are particularly suitable for determining pollutants in a wide variety of products simply and non-destructively.

Table 2 shows examples of detection limits for lead in different materials. It also shows that the required detection limits can also



Pollutant analysis in toys.

be reached for other heavy metals, such as cadmium, mercury, arsenic and barium.

## Measuring the AI coating thickness in the example of an AI/Si wafer

Al coatings occur in various applications in the electronics field. The very thin coating thicknesses and the lack of fluorescence radiation for base materials such as ABS, or the low energy of the fluorescent radiation of the base material Si, make it necessary to take a direct measurement of the Al. With an energy level of approx. 1.5 keV for the Al-K radiation, SDD's high sensitivity offers

Matrix		ABS	A	CuZn
Detection limit Pb/ppm		2	6	50
	RoHS	DIN EN 71		CPSIA
Limit	1,000 ppm	90 pp	m	100 ppm

 Table 2: Detection limit of lead in different materials, measured by the XDVR 

 SDD, compared to the limits specified in various regulations.



FISCHERSCOPE® X-RAY XUV® 773

clear advantages at low energy levels. Figure 1 shows the spectrum of an approx. 1 µm thick Al/Si wafer coating measured with the XDV®-SDD (red line on graph). The fluorescence lines of Al and Si can be seen very well. Because the fluorescence radiation at this energy level is already absorbed strongly by the air, the intensity increases markedly when measured in a vacuum using the





Figure 1: Spectrum of an AI/Si wafer sample measured with  $XDV^{\otimes}$ -SDD and  $XUV^{\otimes}$  773.

«information from practice»

Table 3: Measuring an Al/Si wafer sample with 20s measurement time.

1	Al/nm		
Instrument	Х.	S	
XDV®-SDD	979	11.4	
XUV® 773	953	2.4	

FISCHERSCOPE® X-RAY XUV® 773 (blue line on graph). Table 3 shows the quantitative results on the same sample. The Al coating can already be measured reliably using the XDV®-SDD. However, using the XUV® 773 there is, once again, an improvement in repeatability of more than four times.

Dr Bernhard Nensel

## High Repeatability Precision and Trueness of Au / Pd Coating Measurements on Leadframes

As the electronics industry makes use of ever thinner coatings, manufacturers increase their demands on measuring technologies to provide reliable parameters for product monitoring and control. The coating system Au/Pd/Ni is frequently used in the electroplating of leadframes, with CuFe<sub>2</sub> (CDA 195) as substrate material. Typical coating thicknesses are between 3 – 10 nm Au and 10 – 100 nm Pd. For monitoring the quality of these coating systems, X-ray fluorescence instruments have established themselves as the measurement method of choice.

A series of comparative tests employing other physical measurement methods was used to determine the capabilities of X-ray fluorescence instruments within the mentioned ranges. Sample specimens were measured with the X-ray fluorescence method using the FISCHERSCOPE X-RAY XDV-SDD model, Rutherford backscatter and absolute, synchrotron radiation based X-ray.



Figure 1: High resolution measurement of an Au/Pd/Ni coating system on a leadframe and presentation of the results with the analysis software, WinFTM®.



Figure 2: Lateral coating thickness distribution of a specimen coated with only a few nm Au.

For Au coating thicknesses of about 4, 6 and 9 nm, the results from the X-ray fluorescence instruments were all between the two other methods, with deviations in the sub-nm range, confirming not only the low scatter but also the trueness of measurements using X-ray fluorescence instruments. Traceability of the measurement results is ensured by using the FISCHER calibration standards developed specifically for this measuring application. The simple handling of X-ray fluorescence instruments also allows for easy scanning of a specimen to determine the homogeneity of the coating thickness, if required (see Fig. 2).

The combination of state-of-the-art detector technology and the powerful analysis software, WinFTM®, allows for reliable, accurate measurements of coating thicknesses even in ranges below 10 nm. For use on leadframes, the FISCHERSCOPE® X-RAY XDV®-SDD instruments are recommended for relatively normal-sized specimens; for very small structures, the XDV®-µ model, with its special X-ray optics, ensures a very small measurement spot of only 20 µm on the specimen.



## UK Seminars and Events planned for 2013

#### **Fischer GB XRF Seminar on 1st May**

The first event is a Fischer GB organised Seminar on 1st May. This is a specialist Seminar which aims to train and impart knowledge on XRF technologies and the many practical applications. This will be held at Pure Offices, Sherwood Business Park, Nottingham. To ensure you're on our mailing list for more information, email ccave@fischergb.co.uk or call 01590 684100

#### Fischer GB at IMF Transfair on 11-13th June

Fischer GB will also be supporting the IMF Transfair on 11-13th June at the Heritage Motor Centre, Gaydon. This is a three day conference set to boost transport manufacturing and is ideally suited for Fischer's range of coating thickness measurement gauges, sophisticated probes and XRF instrumentation.



#### Fischer GB 'Classics' Seminar on 9th July

Fischer GB are hosting their 'Classics' seminar at the Heritage Motor Centre. This is a specialist Seminar focusing entirely on the extensive and versatile hand held range of coating thickness gauges, probes and the Fischer Data Centre, a relatively new system which manages much of the measuring process within the transport industry. To ensure you're on our mailing list for more information, email ccave@fischergb.co.uk or call 01590 684100.

#### **Fischer GB at Pipeline Conference** on 15 - 18th October at Aberdeen

Fischer have a range of products specifically developed for the corrosion industry and created much interest at last year's event in Edinburgh, with the demonstration of the prototype FA100® probe for very thick pipeline coatings. Email ccave@fischergb.co.uk for more information.





#### Fischer GB at the British Institute of Non Destructive Testing (BIndt) Conference on 10-12th September

Fischer plan to partake in the conference and exhibit at this specialist Materials Testing exhibition held at Telford on 10-12 September. Please visit us on Stand A13.

#### **Fischer GB at Advanced Engineering on** 12th - 13th November, NEC

Following last year's success, Fischer have booked another stand at this quality exhibition, where a full range of instrumentation will be on display. Please visit us on Stand G25 within automotive and close to the aerospace section.

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