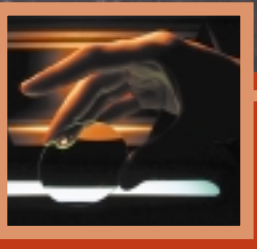
Materials

The Umicore customer magazine | December 2002

umicore
materials for a better life

New name but the same commitment

Optimized targets for LLS EVO



Essilor in the USA

Editor in Chief: Hans Quaderer, General Manager Umicore Materials AG and Ignace de Ruijter, Vice President Umicore Specialty Materials
Managing Editor: Markus Schilling, Marketing Communications Manager
Design Management/ Layout/Production: Denon Corporate Publishing, Rapperswil, Switzerland
Project Manager: REMCOM GmbH, Richterswil, Switzerland
Please contact us at: Fax: +423 388 74 50
E-mail: sales.materials@umicore.com
Internet: www.umicore.com

Going from Raw Material to Finished Product

The overall effect of the Umicore poster campaign – the cover on this issue – is both surprising and humorous, and the first in a series.

By Markus Schilling,
Marketing Communications Manager,
markus.schilling@umicore.com

The new advertising campaign starts with the raw material – and the Umicore concept. Using unexpected pictorial combinations, we have attempted to show the added benefit our company brings to everyday and exotic materials, and how new technologies are developed to enable Umicore to create «materials for a better life». The current series of images is based on the four seasons of a year – spring, summer, fall and winter. The images portray a raw material as it is found in the earth and how it is transformed into a high-quality product, or a whole range of products. Umicore represents the essential link in this development and production process.

The cover of this issue of Materials is our fall motif. The brown material in the background looks like a small road in a rather rough landscape, but in reality is a close-up of zinc ore just as it is mined. Zinc ore is the primary source of germanium, which is used in the manufacture of camera lenses.

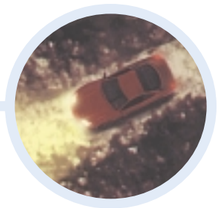
These lenses, in turn, are used in the infra-red night vision systems found in today's high-end automobiles, represented by the small model seen driving across the zinc ore landscape on our cover. Ultimately, this symbolizes the link between the raw material, the application and the final product – the Umicore benefit.

In coming issues, our Materials cover will show other seasons with surprising connections to our technology and products.

We hope you are as surprised and entertained by the unexpected juxtapositions as we were! ●

Zinc Ore.

Lenses.



Infra-red night vision systems.

«2002 turned out to be a success.»

«I am quite impressed by the commitment of the people and the application focus in this business.»

Dear Reader,

It has already been six months since I joined Umicore Specialty Materials and moved from the USA to Liechtenstein with my family. However, beyond the stunning scenery and wonderful local foods, there is the matter of developing, producing and selling coating materials!

Of course, our new business unit is more than the site in Balzers, Liechtenstein – with further sites located in Belgium, Taiwan and the USA. For Umicore, they represent a wholly new business segment and a strategic entry into the thin film materials markets. With their strong background and recognized technology base, I am quite impressed by the commitment of the people and the application focus in this business. On the other hand, the Advanced Materials team also benefits from Umicore's expertise in refining and recycling, the broad product range in non-ferrous metals, and the thin film materials expertise of the High Purity Materials group.

Despite the challenge of integrating the previous Unaxis Materials organization – human resources, information systems and marketing – into Umicore, 2002 turned out to be a very successful year. Overall, we exceeded expectations, with particularly strong revenues in optical data storage – and consistent performance in the other segments that made up for the drop in telecom applications.

For the coming year there will be no surprises. We intend to maintain our focus on the optical data storage, optics and electronics markets. At the same time, we will also intensify our activities for targeted applications such as ITO and high-purity cobalt.

Because every change generates concerns, we have paid close attention to the responses and questions from our customers during the changeover. Initially, there are the practical issues, from the contact person – which remains completely intact – to the more fundamental questions such as, «What is your strategy for the future?» If a concise answer is possible here, I would say that Umicore fully intends to keep the previous focus on quality products and services, while expanding our product range and our worldwide network.

In any case, my editorial team and I sincerely hope you enjoy the «new» issue of our Materials magazine – now beginning its sixth year – with the new layout and colors that reflect the new company and spirit of innovative products and services to come.

To all a very happy holiday season!

Best regards,

Ignace de Ruijter
Vice President
Umicore Specialty Materials
ignace.deruijter@umicore.com

The Name is New, but the Commitment Remains

René Müller: «We've been developing and selling the best materials for PVD over the past 50 years. That's not going to change.»

By Hans Quaderer,
Managing Director,
hans.quaderer@umicore.com

The name change to Umicore brings other changes as well to our PVD coating materials activities – an enlarged global network and a strengthened commitment to growth with a larger, innovative product palette.

This past summer we held a «welcome party» in Balzers, Liechtenstein, to bring together our employees with our new owners from Belgium for the first time, and to celebrate our new name – Umicore Materials AG. My first thought was, «This is a key moment in our 50-year history» and I said so during my short speech. The mood among the employees was light and one could sense the relief now that over a year of negotiations had finally come to a successful end. A number of representatives from the Brussels headquarters of Umicore were also on hand to enjoy the celebration, including Marc van Sande, Executive Vice President of Umicore Advanced Materials, and Ignace de Ruijter, our new business unit manager and also a new Liechtenstein resident. «This is a new beginning for both the Balzers team and myself,» commented de Ruijter. «And I'm looking forward to leveraging the group's impressive technical know-how and market experience to aggressively expand the business in the coming years.»

The best for PVD

While the company name is new, our fundamental know-how and commitment to our customers remains the same.

«We've been developing and selling the best materials for PVD over the past 50 years,» explains René Müller, Production Manager in Balzers. «And that's not going to change.»

What is going to change is more a matter of volume rather than product focus. An important goal is to expand the existing PVD product lines by increasing market share and adding more innovative products. Because Umicore has never had a dedicated activity in coating materials, our know-how and experience opens completely new markets – and opportunities. Umicore is clearly aware of the benefits of adding a portfolio of high-quality products for the optic, electronic, data storage and wear protection market segments; and not only because it ideally complements the company's other activities.

Our «welcome party» in Balzers, Liechtenstein was the perfect kick-off to a new chapter for our company.

The Umicore background

Umicore is the name of what was once called Union Minière, a company with a century-old tradition in the extraction, refining and transformation of non-ferrous metals. The company has shifted from mining and production of commodity metals towards production and marketing of metal-based materials and material recycling.

Committed to a vision of producing «Materials for a better life», Umicore is one of the world's leading suppliers of non-ferrous metals and engineered materials. The way they are actually used is not always visible, but they are found in an amazing range of products. Here are just a few examples:

- Electronic components, solar cells and optical fibers
- Pharmaceuticals, fertilizers and ceramics
- Automobile tires, batteries and roofing materials
- Coins, jewelry and decorative elements

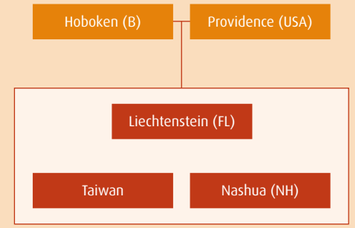
The business of recycling industrial quantities of metals and developing environmentally-sound production techniques in smelting of metals has also grown to become an important part of the Umicore competency. This will also contribute to our know-how.

A global network

As part of the Umicore Group, the Umicore Materials AG in Balzers is part of the Specialty Materials business unit, which belongs, in turn, to the Advanced Materials business group. This business group is one of four that make up the Umicore Group:

- Copper – powder, flakes, billets, rods, and cathodes for the automotive industry
- Precious Metals – extraction of Au, Ag, Pt, Pd, Rh from complex raw materials; recycling industrial scrap
- Zinc – powder for paint, for galvanizing cars, sheet metals for construction (roofs, tubings)
- Advanced Materials – battery products (Co, Ni, Zn, and alloys); engineered powders for hard metals and diamond tools; Co and Ni-based products for chemicals and ceramics; infrared optical materials and components, substrates for electronics and opto-electronics (such as solar cells); GeCl4 for fiber optics; high purity metals (Co, Se, Te, In); and now – Thin Film Materials

Locations around the world

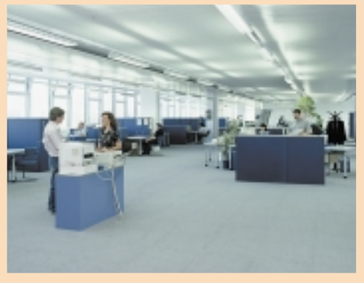


Our network has now expanded to many new locations around the world.

As part of the Advanced Materials group, the Specialty Materials business unit now adds to our global reach, with sites in Hoboken, Belgium and in Providence (RI), USA.

The Thin Film Materials business line is headquartered at the production, distribution and administrative site in Liechtenstein, and now includes our branches in Nashua (NH), USA, and in Hsin-chu Hsien, Taiwan. Both the USA and Taiwan sites function as target bond shops and regional sales offices.

Bursting at the seams at our current location, Umicore Materials will move to a new location – also in Balzers – by 2004.



A computer-simulated view of our new offices in 2004.

New HQ in Balzers

This past summer, we signed a contract for a new production and administration center not far from our current location in Balzers, Liechtenstein. Due to our constant growth in sales and the number of employees during recent years, our current location in Balzers has been much too small for some time. Office and lab space has become so hard to find that individual services such as product development and the warehouse are already located in other buildings in and around the town of Balzers. This makes logistics and certain organizational activities more complex – and costly. ●

Working with Essilor – AR Coatings are Key

Cesar Maksoud: «The North American market for AR coatings is full of potential and Essilor hopes to capitalize on this growing market.»

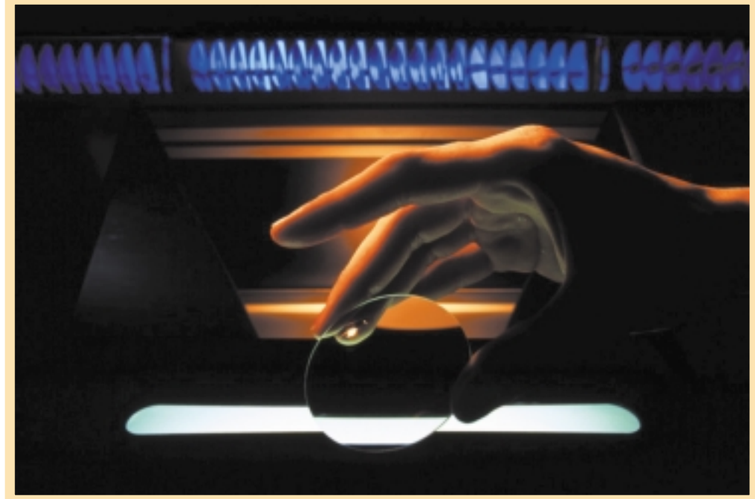
Today's high-performance ophthalmic lenses are synonymous with the name Essilor, a pioneer and the leading manufacturer of progressive and polycarbonate lenses. Essilor of America, Inc. relies on materials from Umicore Materials for deposition of their Crizal® anti-reflective lenses.

By Chaffee Tran,
North America Sales Manager,
chaffee.tran@umicore.com

Essilor of America, Inc. (Essilor) is a subsidiary of Paris-based Essilor International, S.A. Essilor is the leading manufacturer of optical lenses in the North American market and a pioneer in the development and production of ophthalmic lenses. Currently, Essilor is the market leader in progressive, polycarbonate and anti-reflective lenses.

«The North American market for anti reflective (AR) coatings is full of potential and Essilor hopes to capitalize on this growing market,» points out Cesar Maksoud, Director of Coating Technology and Process at Essilor. With over 143 million eyeglass wearers in the United States, Essilor has managed to remain the market leader thanks to research and production of lenses that meet the demands of every lifestyle. One of the unique characteristics of the North American market is the widespread use of polycarbonate (PC) for prescription eyeglass lenses. The purity and stability of PC was improved by compact disc manufacturers to the levels required by the optical industry. But to use this material for corrective lenses, Essilor had to meet three further challenges: mastering a new production process, enhancing all characteristics at the same time (such as weight, thinness, strength, and color), and ensuring consistent performance parameters. Essilor's R&D teams follow a comprehensive «technology watch» process to profit from innovations across many industries. The research staff has produced advances in ophthalmic optics by adopting and adapting technologies from other industries – such as the optical storage disc industry – with seemingly unrelated priorities.

The Essilor of America Inc. headquarters in Dallas, USA.



Essilor continues to add manufacturing capacity to meet the market's growing preference for Rx lenses.

Improving plastic

As a lens material, plastics are far more resistant to impact than glass lenses. However, until the treatments developed by Essilor, they were not sufficiently scratch-resistant, nor could they undergo anti-reflective treatment. These treatments are often more complex and more expensive than the production of the lenses themselves. Essilor, through its proficiency in chemistry (coatings) and manufacturing (thin film layer applications) has acquired a substantial technology lead in this area. Over the years, Essilor has worked on refining and improving the performance parameters for prescription eyeglass lenses (Rx). The continual enhancement of the lens design and lens coatings has helped the company establish a product line that is the most comprehensive of any lens manufacturer. Today, Essilor lens products cover all Rx ophthalmic lens types, from finished and semi-finished single vision, to progressive, multi-focal, high-index and anti-reflective lenses. Essilor's premium product offering includes Varilux® progressive lenses, Crizal® anti-reflective lenses and Airwear® polycarbonate lenses.

«Based on our company's history of leading edge technology and innovation, we are now the leading manufacturer of progressive lenses for ophthalmic lens suppliers in North America and throughout the world,» summarizes Maksoud.

The visible Crizal® difference: The anti-reflective lenses (right) are a market leader.

A major part of the technology offer at Essilor is anti-reflective (AR) lenses. The leading product here is the company's Crizal® AR lens, which combines a three-in-one process to treat against reflections and protect from scratches and dirt. The lens is anti-scratch, anti-reflective and contains a hydrophobic layer on top of the thin film stack.

Maksoud adds, «Our Crizal® product is one of the best AR coatings on the market today.»

A 20-year partnership

Umicore Materials currently supplies AR coating materials for all Essilor lenses. But the Umicore R&D teams have worked together with Essilor for over 20 years for all types of coatings for prescription lens applications. «We have used Umicore's evaporation coating materials since 1982,» recalls Maksoud, who was part of the lens coating team from the start. «And we will continue to use your materials because they offer us the consistency and quality that we require for all our AR products.» Maksoud is optimistic about the future – of the industry and the company, thanks to the steady growth in demand for high-performance Rx lenses in the North American market. Essilor of America expects above-average annual rates of growth for at least the next five years, counting on a relatively high level of investment in R&D to stay on the leading edge of high-performance ophthalmic lenses. «Our cooperation with Umicore – enhancing existing lens coatings and developing the materials for new applications – will remain an essential part of our market strategy,» concludes Maksoud. ●

New Magnet System – and Optimized Targets – for LLS EVO

By Alex Nef (right),
Product Manager Batch Sputtering Systems,
alex.nef@unaxis.com;
and Klaus Leitner, R&D Manager,
klaus.leitner@umicore.com

A new magnet system and a target cooling system for the AKQ 515 cathode in the LLS EVO sputter system from Unaxis Semiconductors achieve dramatic increases in target life and deposition rates. Further enhancements in target design and material properties also contribute to improved system performance.

The LLS EVO is an extremely versatile batch sputter system adaptable to a wide range of applications – advanced packaging, III-V, MEMS, MMIC and optical components, just to name the latest – at very effective «cost of ownership» values. Recent system enhancements to the LLS EVO include:

- New advanced magnet array for higher target utilization
- Improved target cooling for higher throughput
- Optimized sputter targets for the best possible «cost of ownership»
- Special shield coatings to reduce particles and maintenance
- Asymmetric bipolar pulsed DC power supply to improve film properties

Advantages of the advanced magnet system

The new magnet system increases the efficiency of the AKQ 515 sputter source and increases normal target life by at least 50%, even up to 100% or more, depending on the selected target material. During the development and testing of the magnet array, special attention was paid to maximizing the target life while maintaining reproducible film properties over the life of the target.

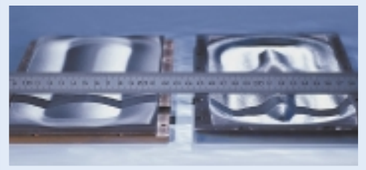
An improved cooling system

The advanced target cooling technology enables doubling of the sputter power to the AKQ 515 sputter source, placing new requirements on the targets. The Umicore targets were optimized and adapted for use with the new cooling system. For example, the new titanium target is a monolithic design replacing the previous bonded type. This allows higher sputter power levels and increases target life even more. The very ductile high purity aluminum and aluminum alloy targets are welded by a special bond process to a stiff copper alloy backing plate, which eliminates the need for a central clamp and allows the use of high power cathodes with higher deposition rates and increased throughput. Overall, the Umicore targets are more compatible to the higher power source – mechanical stability combined with excellent heat dissipation – compared to competitive targets.

An operator at the LLS EVO system.

Process stability

Target life, however, does not tell the whole story about system performance and process stability over the life of the target. The Unaxis Semiconductor R&D team – together with the materials specialists at Umicore Materials – tested all relevant thin film parameters over the life of a target. The results showed excellent stability of the deposition rate, improved sheet resistance and stress. This represents clear net improvements in process stability, even if the extended target life is disregarded. For example, a Ti target sputtered at 550 kWh shows a deposition rate drop of less than 10% (without the rate compensation mechanism built into the LLS EVO systems). The older design target was sputtered at 240 kWh, with a 14% drop in the deposition rate. The enhanced performance of the magnet array improves overall output of the sputter source over previous versions, and about 70% more wafers per target when compared to competitive sources on the market.



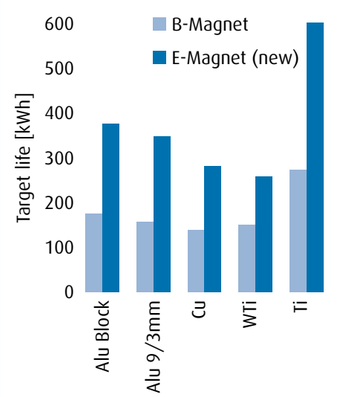
Ti targets at the end of lifetime. Left with B magnet after 220 kWh; Right the monoblock target with E magnet after 550 kWh.

Because the number of wafers processed per target is almost doubled, this reduces the frequency of target changes, resulting in less downtime and maintenance – all contributing positively to a lower 'cost of ownership' and higher yields.

New targets from Umicore

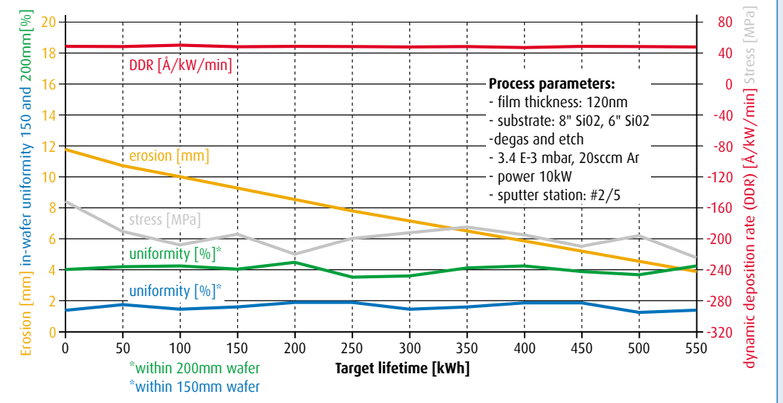
Again, close cooperation between the product teams of Umicore Materials and Unaxis Semiconductors enabled development of an optimized target design that takes full advantage of the higher performance AKQ 515 sputter source. While the new sputter source is suitable for all metal and dielectric materials (except ferromagnetic materials; Ni, Fe, Co and alloys), Umicore redesigned the titanium, 4

Figure 1

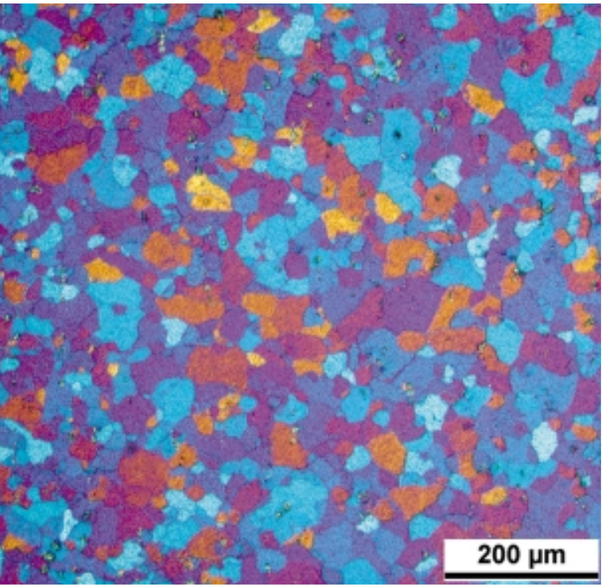


Target lifetime

Figure 2



Ti monoblock target life with E magnet on LLS EVO (sputter source AKQ 515)



Close-up of an AlCu4 microstructure with an average grain size of 20 microns (Barker etching, micrograph in polarized light).

chromium, aluminum and aluminum alloy targets. Thermo-mechanical treatments added to the target production process generate a very fine and uniform globular grain structure. ●

For more information on the LLS EVO system and the AKQ 515 sputter source (and retrofits) please contact alex.nef@unaxis.com.
For more information on the sputter targets please contact bernhard.bracher@umicore.com.

Figure 3

Part Number	Description	Symbol	Purity	Form
BD485095-T	Aluminum	Al	5N5	Compound
BD485121-T	AlCu4	AlCu4	5N5	Compound
BD485122-T	AlSi1Cu0.5	AlSi1Cu0.5	5N5	Compound
BD483078-T	Chromium	Cr	3N5	Monolithic
BD485111-T	Copper	Cu	4N5	Monolithic
BD483869-T	NiV7	NiV7	3N5	Monolithic
BD483075-T	Titanium	Ti	3N	Monolithic
BD483080-T	Titanium	Ti	4N5	Monolithic
BD483843-T	WTi10	WTi10	4N5	Compound

Examples of modified targets

Bridging the CD – DVD Gap

Dieter Dierks: «In fact, DVDplus© could prove to be key to making DVD a mainstream format.»

Not another competing DVD format, but literally two products rolled into one – the DVDplus© combines the familiar CD technology with a high-capacity DVD. A new sputter target material from Umicore has enabled this new format.

By Wolfgang Siegl,
Optical Data Storage Manager,
wolfgang.siegl@umicore.com

The DVDplus© is essentially a CD glued to the back of a DVD5 disc, resulting in a product that can be played on both the huge installed base of over 1.5 billion CD players around the world – and the growing numbers of new DVD players. This allows, for example, a whole concert to be stored as a video recording on the DVD side of the disc, with the same concert available as an audio recording on the CD side.

The new disc comes in different formats:

- DVDplus© – with both film and audio on a single disc
- DVD Audio plus© – combines the conventional CD with the new DVD high-capacity, 24 bit 192k audio format
- DVD ROM plus© – combines film with CD-ROM (for PC games, etc.)

«Because of the access to the endless CD player base, our new format is an excellent promotional lever for the DVD technology,» explains Dieter Dierks, inventor of the DVD-plus© disc. «In fact, DVDplus© could prove to be key to making DVD a mainstream format.» Backers of the new format also point to the disc's potential for collectable editions, and the added capacity to provide room for compilations – enabling the promotion of a «whole back catalog of musical artists in the broadcasting industry».

For example, the Australian FM Mix compilation, featuring artists such as Robbie Williams, Kylie Minogue, Kool & The Gang and The Jacksons, shows how you can compile CD audio tracks and different DVD video clips. The combination of both audio and video on one disc has also caught the interest of the music industry suffering from MP-3 downloading and burning CDs: «The added benefit of DVDplus© with combined film and sound is impossible to copy,» adds Dierks.

For more information on the DVDplus© disc format, please contact Ms. Hatice Aker, h.aker@dvd-plus.de or see on www.dvd-plus.de

Making a DVDplus©

The manufacturing process for a DVDplus© differs only slightly from a normal DVD. In the initial stage, two molds have to be produced for the DVDplus© – one for the CD side and another for the DVD side. In the duplication process, the compact disc, with a slightly reduced thickness, is bonded to one side of a DVD5 disc. The replication costs for a DVDplus© disc are similar to a DVD9, which is used widely for pre-recorded TV and movie videos.

There have been questions about the DVDplus© meeting the Red Book specifications, mainly because of the disc thickness. A laser will read through a DVD5 (0.6-mm thick) without a problem and it will also read through a normal CD (1.2-mm thick) with equal ease. However, Dieter Dierks has developed a disc 1.48-mm in width, and demonstrated playability on 100% of all CD, DVD and computers used in the phase one testing done so far. Further rigorous tests through an independent source, commenced in November 2002, are expected to confirm these results. Visually, the DVDplus© is recognizable by its two playable sides, similar to a DVD10 disc in appearance. This means the surface can only accommodate a very limited amount of printing. While this is a present limitation, Dieter Dierks is already developing a new template to enable a larger area to be utilized for printing. DVDplus© is gaining momentum in most sectors as content owners begin to understand the versatility of the format. It is now widely used throughout Europe, with sales of more than 1,8 million with focus on Germany, highlighting a triple platinum award for DVDplus© release of Herbert Grönemeyer, with a good number of key releases in Australia and now gaining immense interest in the USA. «Music companies and film companies are acknowledging that DVDplus© is not a competitor to DVD or CD, but is the perfect bundling solution for a new front line product or for repackaging back catalogs,» concludes Dierks. ●

The Australian FM Mix compilation is a popular DVDplus© offering.

Thermal Materials Analysis

Working with the STA 449 C Jupiter® instrume nt from NETZSCH

Dr. Fritz Waibel: «This instrument is able to provide relevant data for a very wide variety of materials.»

The breathtaking innovative pace towards smaller and smaller dimensions and higher component performance is not limited to the computer industry. Increasingly consistent production and product quality standards for optical evaporation materials and electronic sputter targets have been implemented, thanks to our thermal analysis capabilities.

By Andreas Hiermer,
R&D Project Manager Optics
andreas.hiermer@umicore.com

In order to keep pace with increasing market demands for higher performance products, our materials analysis lab in Balzers, Liechtenstein, depends on two key aspects:

- Precise measurements of the materials used in our products
- Adaptable measurement hardware

The new STA 449 C Jupiter® simultaneous thermal analysis instrument from NETZSCH meets our expectations – and has notably enhanced our lab's analysis capabilities since its installment in early 2002.

The STA 449 C Jupiter® thermal analysis system enables simultaneous observation of weight behavior and the DSC (differential scanning calorimetry) signal on one and the same sample under identical conditions. This means the mass changes of a sample during heating and the caloric effects can be accurately and comprehensively measured at the same time. With a maximum sample weight of 5 grams, which also represents the weighing range, the STA 449 C Jupiter® achieves a resolution of 1 µg. Operating the system is literally as easy as filling in the material, closing the lid and pushing a button. Best of all, the analysis know-how is in the system and in the evaluation of the results. With easily exchangeable furnace systems and the various application-specific sensors, almost every imaginable application can be analyzed in the temperature range between 20°C and 1650°C. This also includes determination of the specific heat over the entire temperature range with high precision and reproducibility.

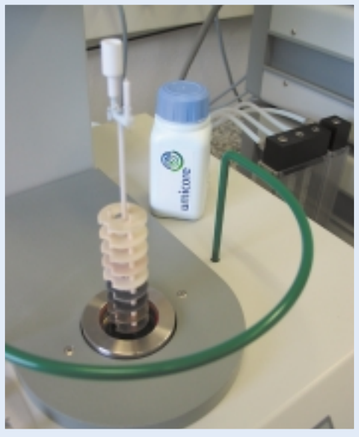
Versatile – to the decimal point

Dr. Fritz Waibel, the chemist at the lab states: «Our thermal analysis system is very versatile. This instrument is able to provide relevant data for a very wide variety of materials. Many of our products need to be repeatedly sintered at precisely defined conditions. These we determine with our new instrument within a reasonably short time, which helps us to adapt the material processing parameters – to the decimal point!»Characteristic for the thermal analysis instrument are the extremely stable and reproducible TG (thermo-gravimetric) and DSC baselines. The DSC sensor's high degree of sensitivity is guaranteed over many hours, even at temperatures above 1500°C. The electro-magnetically compensated, top-load ultra-microbalance is remarkable due to its high accuracy and resolution in the sub-mg range, as well as its excellent stability. These features underline the superior and unique performance of this new thermal analysis instrument.

Imperative research tool

The vacuum-tight design of the STA 449 C Jupiter® allows coupling with a gas analysis system – such as a quadrupole mass spectrometer and a fourier-transformed infrared spectrometer. This provides the capability for a practically complete characterization of a sample with a single measurement by combining thermal analysis and gas analysis. A further feature is the dilatometer also provided with the instrument. This module monitors the dimensional changes of the materials as a function of temperature or time, while the sample is subjected to a minimum mechanical load.

«The high precision and excellent reproducibility make it an imperative tool for research and development of quality products at our lab,» adds Waibel. «We've managed to halve the time for product development thanks to the elimination of a large number of test runs that were previously needed to get the same results.» ●



Placing a sample in a ceramic crucible on the measuring head.

Table1

Method	Guideline	Definition
DSC	ICTA, 1999	Differential Scanning Calorimetry monitors the heat flow rate to the sample against time or temperature, while the temperature of the sample, in a specific atmosphere, is programmed.
DTA	ICTA, ASTM E-473-85	Differential Thermal Analysis measures the temperature difference between the sample substance and the reference material as a function of temperature, while the substance and the reference material are subjected to a controlled temperature program.
TGA	ICTA, ASTM E 419-83	Thermo-Gravimetric Analysis measures the mass of a substance as a function of temperature or time, while the substance is subjected to a controlled temperature program.

Summary of the various types of thermal analysis for coating materials.

Table 2

Temperature range:	20°C – 1 650°C
Thermogravimetry:	
Sensitivity	0.1 mg
Sample weight	max. 5 g
Measurement range	0 – 5 000 mg
Isothermal drift	<1 mg/h at 1 200°C
Calorimetry:	
Sensitivity	Dependent on the sample carrier system used (e.g. 18 mV/mW) at the melting point of indium.
Measurement range	500 – 5 000 mV
Sample atmosphere:	– Static/dynamic/ vacuum – Oxidizing/inert – Vacuum approx. 1 x 10^{-4} mbar

Technical data of the STA 449 C Jupiter®

Operating the thermal analysis instrument at the Balzers lab.

Density-Related Properties of Metal Oxide Films

The density of a film material determines many other important film properties. For instance, high density decreases the film's permeation property and enables the production of barrier layers against diffusion of gases, vapors and liquids, and also against diffusion of solid atoms and ions.

By Dr. Hans Pulker,
University of Innsbruck,
Austria

Concerning mechanical film properties, low density values result generally in tensile stress, whereas high-density values generate compressive stress. In both cases bending in one or the other direction deforms thin substrates. Very high values influence adherence and may even cause local delamination or blistering in the case of compressive stress and cracking due to tensile stress. Surface hardness and abrasion resistance of a film are generally increased with compressive intrinsic stress.

Concerning optical properties, the refractive index of a film is strongly influenced by the density of the material. High density results in high refractive index and low density decreases the value. Very high densities seem also to lower the resistance of chemical compound films against color center formation by local non-stoichiometry when exposed to short-wave radiation.

The final film properties can be strongly determined by the right production technology, the chosen parameters, and by special post-deposition treatments.

Film formation and properties

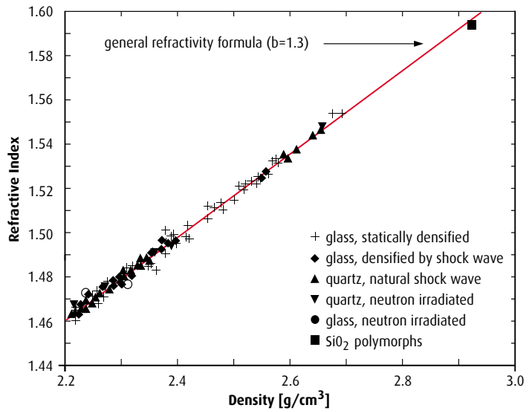
Chemical compound films such as metal oxides and some nitrides and oxynitrides are mainly produced by reactive PVD processes. For films used in precision optics, ion and plasma-assisted technologies are preferred because of their high chemical and physical activity and the possibility of depositing onto unheated substrates.

Such films obtained under high energy coating conditions are generally amorphous and in meta-stable constraint high-density states. They have a homogenous uniform microstructure and a smooth topography. Stoichiometric films are practically free of optical absorption in their high transmittance region. Typical k-values are below 10^{-5}.

Concerning the refractive index of an amorphous material, a strict relationship between density and refractive index exists according to a general refractivity formula [1] based on the Lorentz-Lorenz equation and on the Newton-Drude formula. An increase in density increases also the refractive index as can be seen in Figure 1. A high energy input into the growing film results in both high density and a high refractive index.

However, the high density also creates a high compressive film stress, positively influencing hardness and abrasion resistance, but negatively influencing flatness and adherence to the substrate and between films in multi-layer coatings. The stress bending moment generally increases with film thickness and stress produces further birefringence in the film. Very high-density values may even create bond length and bond angle deformations, which stimulate localized defect formation, for example, when irradiated with energetic photons. Oxygen vacancy centers and non-bridging oxygen hole centers, responsible for optical absorption are formed in densified fused silica and silica films (such as under irradiation of deep UV excimer laser light, λ = 193 nm ArF, λ = 248 nm KrF) more easily than in low density material. But irradiation not only produces defect centers, it also may induce defect recombination processes. The dominance of one or the other process depends on material parameters and exposure conditions [2].

Figure 1



Plot of refractive index n versus density r of differently prepared SiO_2 samples using the linearized form of the general refractivity formula:

$$\frac{4\pi}{n^2-1} = \frac{1}{\alpha/M}\frac{1}{\rho} - b \quad \text{(linearized)} \qquad \frac{n^2-1}{4\pi + b\,(n^2-1)}\frac{1}{\rho} = \alpha/M \quad \text{(general refractivity formula)}$$

Taken from a publication of Arndt and Hummel (1988) [1].
α = molar polarizability $cm^3\ mol^{-1}$; M = molecular weight g mol^{-1}; ρ = density g cm^{-3}; α/M = constant = 0.0364 $cm^3\ g^{-1}$ and b = 1.3. Relaxed fused silica is the reference point in the graph.

Table 1: Refractive indices of differently prepared TiO_2 films.

Film Material	TiO_2				
Deposition Method	CRE*	RIBAD*	RLVIP*	RPMS*	
Refractive Index				unipolar	bipolar
n_{550}	2.40	2.46	2.55	2.52	2.65
n_{633}	2.30	2.43	2.51		

Refractive indices of differently prepared TiO_2 films. The dense films with the higher index values also show a higher hardness and a higher abrasion resistance. [3, 5-7].
*See Table 2 for explanation of abbreviations.

Table 2

Deposition Method	Film Stress(MPa) SiO_2	TiO_2	Ta_2O_5
Conv. reactive evaporation (CRE) d~250nm	-500	255	150
Reactive ion beam assisted deposition (RIBAD) O^+/O_2^+, 75-120 μAcm^{-2}, 30V d~300-400nm	-1100	-540	
Reactive ion beam sputtering (RIBS) d~300nm	-900	-400	-450
Reactive dual ion beam sputtering (RDIBS) Ar^+, O_2^+, 100 μAcm^{-2}, 500V d~300nm	-700		-600
Reactive pulsed magnetron sputtering (RPMS) unipolar: (RPMS) bipolar: d ≥100nm		-100 -250	
Reactive low voltage ion plating **RLVIP** d~200nm	-700	-800	-700

Mechanical stress of various metal oxide films deposited with different reactive deposition methods by using oxidic starting materials [3-7] (- = compressive stress).

Results

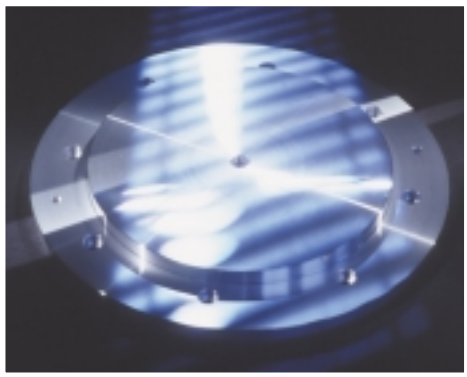
Conventional reactive evaporation of metal oxides from oxidic starting materials yields generally environmental non-stable films with columnar microstructure and low density. The films have relatively low extinction values, but compared to bulk properties also lower refractive indices and exhibit tensile mechanical stress.

Energetic coating processes, however, result in amorphous higher density films of comparably high refractive indices (see Table 1), independent of the applied metal or oxide staring material. They may show sometimes slightly higher residual optical absorption and have relatively high compressive stress values (see Table 2). Longer lasting post-deposition heat treatments of 4 hours at 350°C on atmosphere start relaxation, recombination and possibly oxidation processes, resulting in a slight decrease of film density and refractive index, but in a remarkable reduction of the optical losses and of the compressive film stress values. After such a heat treatment, the physical film thickness is increased slightly due to the decreased density. However, no recrystallization was observed after heat treatment. The films remain in a dense amorphous state and absorb no water vapor at humid atmosphere. Such dense and abrasion-resistant films with environmentally stable optical properties, but with low compressive stress, are in demand for the production of high quality optical thin film components. ●

References:

[1] J. Arndt, W. Hummel, Phys. Chem. Minerals 15 (1988) 363-369
[2] J. Moll, Photonics Spectra, (April 2002) 78-81
[3] H. Selhofer, E. Ritter, R. Linsbod, Applied Optics 41 (2002) 756-762
[4] G.N. Strauss, Chapter 9 in Optical Interference Coatings, N. Kaiser, H.K. Pulker (Eds.), Springer, Berlin (in press)
[5] H.K. Pulker, Coatings on Glass, second revised edition (1999) Elsevier, Amsterdam
[6] U. Krause et al., Proceedings 4th International Conference Coatings on Glass ICCG-4, Gläser, Klager, Aegerter (Eds.), Braunschweig (2002) 125-128
[7] P. Frach et al., Proceedings 4th International Conference Coatings on Glass ICCG-4, Gläser, Klager, Aegerter (Eds.), Braunschweig (2002) 77-81

Al Alloy is not Al Alloy!



Our aluminum targets produce reliable results for both CD and – more importantly – for DVD-9 production because the characteristics of the AlMg-Si/Mn alloy are optimized for the metallization sputtering process. Manufactured according to our specifications, the AlMg-Si/Mn alloy is not a «technical Al alloy» from an undefined production process for Al alloy rods – as widely supplied to the price-driven ODS market by our competitors. Our Al targets differ from lower cost competitive products because the complete production process – from base metal to packaging of the finished target – is exactly defined and controlled by our process-orientated QMS. Beginning with the base material, alloy casting includes a filter step to eliminate non-metal particles such as oxides – which might easily cause sudden energy discharges and lead to disruptions in the thin film layer – during the sputtering process. A heat treatment step guarantees a homogenous structure. Alloy element precipitation is finely dispersed. Compared to standard Al-alloy production, this method assures the end-user consistent process quality and the best possible thin film characteristics – essential for full reflective layers in DVD-9 because of different wavelength, corrosion resistance requirements and specifications when compared to CD. ●

For more information on our Al-alloy targets please contact wolfgang.siegl@umicore.com

Trade shows January – April 2003

Optics		
OFC	March 23 – 28	Atlanta, USA
Optical Data Storage		
Replication Asia	March 26 – 28	New Dehli, India
Media-Tech Showcase/Conference	April 13 – 15	Frankfurt, Germany
Wear and Decorative Coating		
WDC Conference	March 17 – 18	Venice, Italy
ICMCTF	April 28 – May 2	San Diego, USA
Semi/Electronics		
Semicon China	March 11 – 13	Shanghai, China
Semicon Europe	April 1 – 3	Munich, Germany

Bond shop to open in USA

A new bond shop is now operational in Nashua, NH. Initially, the shop will focus on the optical data storage market segment – Si targets for both Unaxis and Singulus metallizers used for DVD9 production. Ultrasonic testing equipment will assure that every bond meets or exceeds industry standards. The shop plans to expand quickly to be able to service semiconductor applications.

«Improved service and quicker turnaround for our customers are the main reasons behind the new bond shop,» explains Bill Reeves, Sales Manager for the North American region. «Local service is key for maintaining further growth in this market for Umicore.»

Further enhancements include a sputtering system for deposition of the thin film coats as diffusion and wetting layers for bonding to indium – to be installed soon. ●

Contact bill.reeves@umicore.com for more information on the new bond shop.