Tarnów Construction Plastics. Significance of construction plastics for the Azoty Tarnów Capital Group

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Azoty Tarnów Capital Group was established in 2010 ÷ 2012 as a result of a consolidation of the so-called Great Chemical Synthesis Branch carried out by Zakłady Azotowe in Tarnów-Mościce S.A. The group's chemical production is concentrated in four companies:

- Zakłady Azotowe in Tarnów-Mościce S.A.
- Zakłady Chemiczne Police S.A.
- ZAK S.A.
- ATT Polymers.

The scale of operations currently undertaken by Azoty Tarnów is illustrated by basic financial data. In 2011, the profits of the Group equalled approx. PLN 5.3 billion, while the net profit equalled PLN 499 million. At the end of 2011, equity capital reached the level of PLN 3.1 billion.

Figure 1 shows the sales structure of Azoty Tarnów in the first quarter of 2012. During this period, construction plastics produced in Tarnów and in Guben constituted the third product group of the company after nitrate and multi-component fertilisers, with 13.4% share.

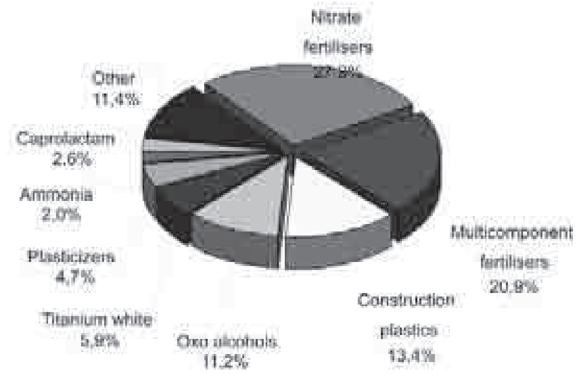


Fig. 1. Sales structure of Azoty Tarnów in the first quarter of 2012

This share is the result of a large increase of the sales of plastics over the last years, especially of polyamides. Figure 2 shows a change in the sales volume of construction plastics in Azoty Tarnów in 2001÷2011.

Such large sales increase was possible thanks to a package of modernisation investments carried out in Tarnów-located Polyamide, Polyoxymethylene and Modified Plastics Plants as well as thanks to the acquisition of the German company Unylon Polymers GmbH.

The development strategy adopted for 2012÷2020 assumes that, similar to 2007 ÷ 2011, construction plastics will constitute one of three elements of domain of the Azoty Tarnów Capital Group, next to mineral fertilisers and chemicals.

In accordance with the aforementioned strategy, polyamides, polyoxymethylene and modified plastics will constitute key elements of the Group's product portfolio in the segment of construction plastics. In the nearest years, an increase of the production capabilities of construction plastics as well as further product diversification in this field are anticipated.

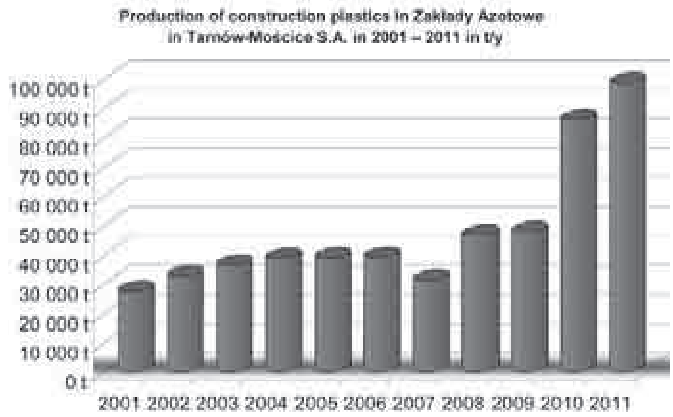


Fig. 2. Change of sales volume of construction plastics in Azoty Tarnów in 2001 ÷2011

The sector of technologically advanced materials, including construction plastics, will also be one of the key fields of focus with regard to the research activities of Azoty Tarnów. From the geographical point of view, Europe is the Group's target market in the construction plastics sector.

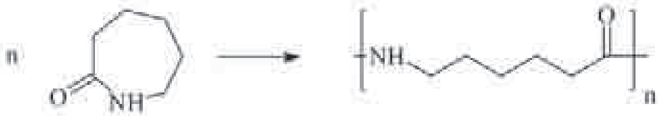
Scope of offered products

Construction plastics are materials characterised by especially good mechanical properties. Among the construction plastics produced by Azoty Tarnów Capital Group are polyamide 6 with the trade name of Tarnamid® and acetal copolymer (POM) with the trade name of Tarnoform®, as well as modified plastics.

Polyamides are multimolecular compounds containing recurring -CO-NH- amide bonds in the molecule. Polyamides hold the top position among construction plastics.

Polyamide 6

Polyamide 6 is produced through condensation polymerisation of caprolactam, occurring according to the following pattern:



Polyamide 6 has a number of very favourable physical properties, including: high mechanical resistance, rigidity, hardness, high resistance to scratches and chemical resistance, especially to organic solvents. Thanks to its properties, it is broadly applied in many fields, including in: the automotive industry, electrical engineering, machine-building industry, construction, furniture industry, sporting goods manufacturing, household appliances, and in the production of polyamide fibres and packaging.

The history of polyamide in Zakłady Azotowe in Tarnów-Mościce S.A. started in the 1950s. In the second half of the 1950s, an installation was constructed and put into operation, based on the technology and supplies from the German Democratic Republic, with an output of 4,000 tons/year, for producing caprolactam and later polyamide 6 on its basis. Over the years, the installation's capacity was slowly increased by constructing further production sequences. Polymerisation was conducted in periodically operating reactors with a capacity of approx. 3 m^3 each.

At the end of the 1970s, the outdated polymerisation installation was modernized, including in the operation a further, seventh polymerisation unit of a newer generation, fitted with an agitator and creating products with a better and repeated quality. The periodic production of polyamide 6 in the 1970s reached the capacity of approx. 6,500 t/y.

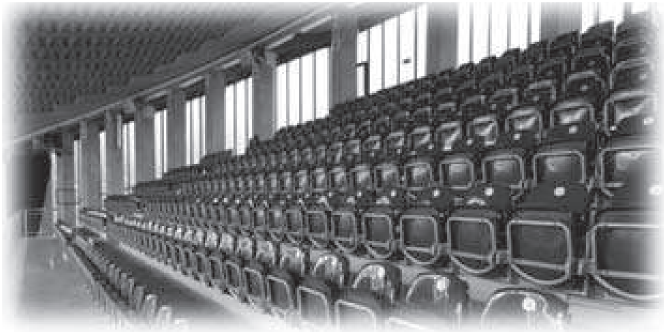


Photo 1. Polyamide 6 seats in sports facilities

At the same time that polyamide 6 was produced using condensation polymerisation, it was also created using anionic polymerisation. This way was used to obtain polyamide 6 in the form of blocks with the trade name of Tarnamid® B, meant for producing details using mechanical processing. The production of Tarnamid® B finished at the beginning of the 1990s.

In June, 1996, after record-short construction and assembly works, a modern installation was started for the polymerisation (polyaddition) of caprolactam, with an output of approx. 23 ths t/y, which continuously produced polyamide Tarnamid® T-27 standard and highly-adhesive Tarnamid® T-30 according to a license purchased in the German company Zimmer. The perfect, stable product with a repeatable quality soon made its way to the world markets, joining the already known caprolactam and ammonium sulphate. The worn out installation of periodic polymerisation, being in operation since 1957, was put out of operation and its devices and fittings were disassembled.

The plant put into operation in 1996 consists of two basic installations, i.e. a polymerisation installation and an installation for recovering caprolactam from post-extraction waters. Polymerisation is a continuous, one-stage process occurring in one VK tube reactor with three internal heat exchangers. Depending on the set parameters, the process may lead to obtaining a polymer with a relative viscosity in 96% H_2SO_4, within the scope of 2.4 – 2.9. Such produced granulate is subjected to a two-stage extraction with hot water to the content of the monomer and low-molecular oligomers not exceeding 0.5%. The second stream is directed to the third extraction level in which the content of monomer and oligomers is lowered to 0.3%, and then to the secondary condensation reactor in a solid state where, in the nitrogen stream with high temperature, the molecular mass of the initial polymer with a relative viscosity of 2.4 – 2.9 is increased to a viscosity of 3.0 – 3.8 depending on the viscosity of the initial polymer and the temperature of nitrogen.

In 2004, this installation was intensified to 27,000 tons/year, while in 2007 it underwent thorough modernisation, changing the polymerisation technology from one- to two-stage and increasing its production output to 45,000 t/year acc. to the license of the Zimmer company.

Photo 2. Installation of B – 170 polyamides

In 2008, Zakłady Azotowe in Tarnów-Mościce S.A. made its debut on the Stock Exchange. One of the objectives of the share issue was to double the production of polyamide 6 in order to balance the production of caprolactam and polyamide and to extend the production chain and limit the sale of raw materials for the benefit of the above-processed products.

As early as in 2008, an opportunity arose to purchase the German polyamide 6 plant – UNYLON Polymers GmbH in Guben – which was in the state of insolvency. The favourable location of the company and suitable production capacity, i.e. 46,000 t/year, were an additional advantage of the project. Following many analyses and negotiations, the purchase of 100% shares of that company took place in December, 2009, thus increasing the production capacity with regard to polyamide 6 up to approx. 91,000 tons/year.

After taking over by Azoty Tarnów and carrying out a corrective program, the production plant in Guben, under the new name of ATT Polymers GmbH and with a new product name of alphalon®, resumed normal production and regained the lost markets.

The plant in Guben produces polyamide 6 for three market segments, i.e. technical and carpet fibres, packaging foils especially for food products, as well as construction plastics. The plant has five sequences of a two-stage, continuous caprolactam polymerisation with a possibility to produce polymers with a relative viscosity range of 2.4 – 4.0, working based on the license of UHDE Fischer. Three sequences work based only on pure caprolactam, while the remaining two process regenerated caprolactam from all lines.

Table 1

List of polyamide 6 polymers offered by the Azoty Tarnów Capital Group

Name of polyamide 6 grade	Relative viscosity of the solution	Water content, %	Content of extracted substances, %	Content of free amine groups, μeq/kg
Tarnamid® T-27	2.65-2.75	≤0.1	≤0.6	
Tarnamid® T-30	3.80-4.00	≤0.08	≤0.3	
alphalon® 24	2.35-2.45	≤0.1	≤0.6	
alphalon® 25	2.45-2.55	≤0.1	≤0.6	
alphalon® 27	2.67-2.73	≤0.05	≤0.6	34.5-39.4
alphalon® 27C	2.60-2.80	≤0.1	≤0.6	32.0-40.0
alphalon® 27D	2.67-2.73	≤0.1	≤0.6	32.0-40.0
alphalon® 32	3.15-3.25	≤0.06	≤0.6	
alphalon® 33	3.25-3.35	≤0.06	≤0.6	
alphalon® 36	3.55-3.65	≤0.06	≤0.6	
alphalon® 40	3.95-4.05	≤0.06	≤0.6	
alphalon® 40H	3.90-4.10	≤0.06	≤0.6	

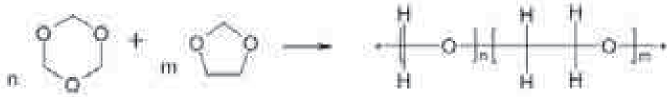
alphalon® grades are offered also in modified form, in which case they have an additional denotation: L – contains lubricant; N – contains nucleant; T – contains thermal stabilizer.

Currently, preparations are being made in the Guben plant for further intensification. In the nearest years, the production capacity should be increased up to approx. 55,000 t/year, mainly in the segment of granulation for technical and carpet fibres. Moreover, further development is planned of the laboratory and research facilities for highly adhesive polyamide meant for producing packaging foils.

Original Polish polyacetal with the trade name of Tarnoform®

The second polymer offered in Azoty Tarnów Capital Group is an original Polish polyacetal with the trade name of Tarnoform®.

Tarnoform® is an acetal copolymer, obtained as a result of copolymerisation of trioxane (cyclic formaldehyde trimer) with comonomers from the group of cyclic oxides (ethylene oxide, dioxolane, dioxepam, tetrahydrofuran, etc.) acc. to the following general pattern:



The technology for producing Tarnoform® was developed thanks to the cooperation of three key entities: Zakłady Azotowe in Tarnów-Mościce S.A., the Fertiliser Research Institute in Puławy and the Industrial Chemistry Research Institute in Warsaw. Cooperation and support was provided also by the Centre for Molecular and Macromolecular Studies of the Polish Academy of Sciences in Łódź, the Institute of Machines Construction and Exploitation at the Wrocław University of Technology, the Institute of Mechanics and Machine Design Basics of the Cracow University of Technology, the Institute of Heavy Organic Synthesis "Blachownia", the Institute of Plastic Working of Metals and Plastics at the Częstochowa University of Technology.

The technology, present in numerous Polish and foreign patents, includes the synthesis of monomers, their polymerisation and processing of a raw polymer to a granulate for producing finished products using injection or moulding or for further processing through compounding.

Works on obtaining Polish polyacetal started in the 1960s.

The creation of a modern polyacetal production plant in Zakłady Azotowe in Tarnów brought about the necessity to select the best possible solution among alternatives at each stage. The question was raised of what type of polyacetal – homopolymer or copolymer – should be produced. Initial trials were conducted on obtaining homopolymer but in the end a decision was made to use a copolymer of 1,3,5 – trioxymethylene.

The first step involved obtaining formalin from methanol on a technical scale. Two methods were tested: with a silver catalyst in the existing installation in Tarnów and with an oxide catalyst in an installation built especially for this purpose. The silver method was chosen because of a better familiarisation with the process.

The next stage involved obtaining monomers of 1,3,5 – trioxymethylene called trioxane and dioxolane.

The synthesis of trioxane was tested using two methods: with mineral acid and ionites. Each of them required the development of suitable solutions with regard to the instruments and the manner of carrying out the process. Both methods involved the selection of suitable instrument materials, either resistant to acidic environment or eliminating corrosive impurities unfavourably influencing the catalyst, as well as the selection of optimum conditions for carrying out the process. Due to lower investment costs, the method involving acid was selected. As opposed to the method involving ionite where it is necessary to use different instruments for synthesis, distillation as well as pumping devices, in the method with acid the reaction and distillation are carried out using the same instrument. The isolation of trioxane from synthesis products may be performed through extraction or crystallisation. The manner of extraction popularized in the world had certain disadvantages related to maintaining the hermeticity of the system and was burdensome for the environment; thus, solutions were developed in Tarnów which allowed for obtaining trioxane through crystallisation. The development of crystallisation systems proceeded from horizontal crystallizers with radial scrapers, then with deflected scrapers, through vacuum crystallizers tested on an experimental scale, until the crystallisation systems used currently on a technical scale, with a tank and scraper crystallizer, which constitutes an original Polish solution. This method allowed for obtaining a trioxane crystal with very high purity, which considerably simplified further purification of the monomer through distillation. At first, the final purification solution was periodically two-stage, while later it was substituted with a two-stage continuous process. Many problems occurred during the storage of trioxane, out of which one very important phenomenon was the creation of a high-molecular polyoxymethylene on the instrument's walls and the creation of impurities from trace amounts of formaldehyde. As a result of the carried out improvements, a considerable decrease was achieved of the impurities in trioxane which impact the polymerisation process. Moreover, new possibilities were obtained in the further process, especially the possibility of lowering the amount of catalyst and of introducing other modifiers of the polymerisation process.

The second monomer, dioxolane, was initially obtained using the periodic method from formaldehyde and ethylene glycol in the presence of mineral acid and the obtained product contained many impurities; it was necessary to neutralize the acid and the entire process was harmful to the environment. Alternatively, a periodic method was tested on an experimental scale of the reaction of trioxane with ethylene glycol in the presence of ionite, in which the flaws of the previous method were eliminated to a large degree. Finally, a two-stage solution was offered – a periodic process of the reaction of formaldehyde with ethylene glycol to semiacetal, and then its conversion to dioxolane in the presence of acid in a continuous process. The obtained monomer is then purified chemically and through distillation.

Together with works on obtaining monomers, a technology of polymerisation and processing of raw polymer was being developed. Initially, periodic solvent and precipitation polymerisation was carried out in a cyclohexane environment. The process using this method proved to be very unstable, while the obtained polymer was not repeated in subsequent batches of the material. The next generation involved continuous, solvent-free polymerisation in a monomer melt with the presence of molecular mass stabilizers and a reaction catalyst. Among various solutions regarding instruments, a reactor was selected based on a double screw extruder, with a suitable construction of screws and a special solution regarding their drive. The raw polymer in the form of a powder was then subjected to processing – size reduction, deactivation of the catalyst, drying and stabilisation, and then granulation. With regard to mechanical properties, the first generation of the Tarnów polyacetal reached the level of approx. 90% compared to foreign polyacetals. Unfortunately the disadvantage involved arduous smell emitted from formaldehyde which became an area of further improvement. As a result of works, the granulation process was combined with a thermal method of removing the so-called unstable fraction in an air stream, while further research and development works allowed for improving the stabilizing compositions and for considerable improvement of the quality of the obtained Tarnoform®.

Many unique solutions were applied in the production technology of Tarnoform® with regard to environmental protection. Here, one may mention the return of formalin and trioxane to the process, the use of a low-energy pressure concentration of formalin, treatment of formalin waste, incineration of waste gas and combustible sewage with energy recovery, or purifying air from impurities such as formaldehyde and catalytic incineration of impurities in the air from the processing of granulate.

The construction of the plant on a technical scale started at the end of the 1980s. The start-up occurred in 1994. Two years later, the designed output was achieved of 5,000 t/y of Tarnoform® for injection processing, while in 1997 an output of 10,000 t/y was achieved and the range of Tarnoform® was expanded by grades for pressing. After modernisation in the first decade of the 21st century, the production capacity was increased by 30%.

The production plant of Tarnoform® includes seven installations – formalin, formalin concentration, trioxane, dioxolane, butylal, polytrioxane as well as combustion of waste gas and liquid – employing nearly 100 people together with the technological staff.

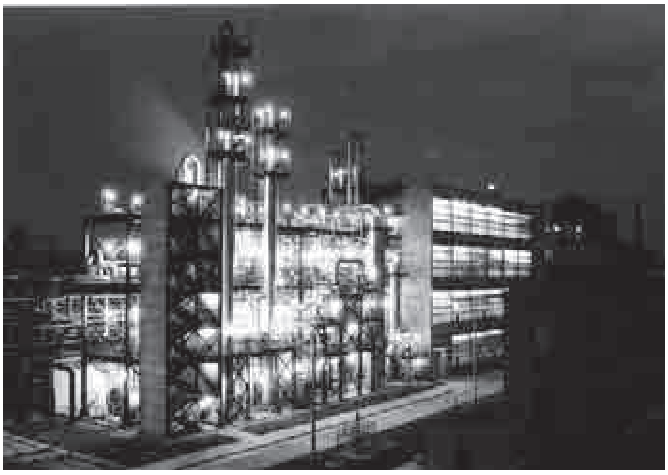


Photo 3. Tarnoform® installation

The achieved level of technology with regard to the product quality, use of raw materials and energy, as well as environmental protection constitutes a competition against the technology of other renowned manufacturers of polyacetals. For this reason, Polish manufacturing technology of polyacetal has become the object of export to Taiwan, North Korea and China.

Table 2

List of polyoxymethylene polymers offered by the Azoty Tarnów Capital Group

Name of polyoxymethylene grade	Melt flow rate (MFR), 190°C/2.16kg	Melting point, °C
Tarnoform® 200	2.5	167
Tarnoform® 300	9	167
Tarnoform® 400	13	167
Tarnoform® 411	13	172
Tarnoform® 500	27	167

Photo 4. Speaker, lock and joints of compounded plastics

Basic polymers of Tarnamid®, alphalon® and Tarnoform® currently constitute the basic offer for customers and, at the same time, are the fundamental raw material basis for producing modified plastics in the compounding technology.

The history of modified plastics in Zakłady Azotowe in Tarnów-Mościce started in the 1970s. In the 1990s, the modification of Tarnamid® and Tarnoform® was continued on a small scale, while since 2001 the process of compounding development started based on the most modern production lines fitted with concurrent double-screw extruders and gravimetric dosing devices for polymers as well as additives, ensuring stable product quality and fulfilling the strictest requirements set by customers.

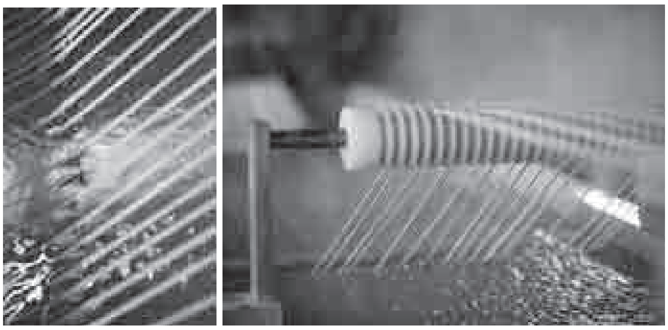


Photo 5. Compounding

Compounding is the physical process of mixing modifying admixtures in a melt/plasticized polymer. The process requires relatively low financial outlays. The compounding process offers the possibility of obtaining composites which broaden the use of polymers in applications unreachable for basic polymers. The most often used additives are so-called staple glass fibres, glass marbles, mineral fillers, nanoadditives, impact modifiers, flame retardants, thermal and UV stabilizers, delustrants, additives facilitating processing, and dyes providing the desired colour and aesthetics of the surface. Compounding fulfils an important role in the process of delivering plastics to end manufacturers. Extending product chains favourably impacts the shaping of added value and the availability of a broad range for customers.

Compounded plastics are meant for the automotive industry, electrical engineering, machine parts, household products, sporting goods and other.

The current offer of modified grades includes a range of product groups:

- Modified grades of Tarnamid:
- reinforced with glass fibre 10% to 50%
- grades with glass marbles 10% to 40%
- grades with mineral fillers 5% to 40%
- hybrid types, e.g. with glass fibre, glass marbles and/or minerals
- grades with halogen-free flame retardants
- grades with increased impact-resistance
- UV light stabilised grades
- high-temperature stabilised grades up to +150°C
- tribological grades
- coloured grades

Modified grades of Tarnoform:

- reinforced with glass fibre 10% to 40 %
- grades with glass marbles 10% to 30%
- grades with increased impact-resistance
- UV light stabilised grades
- tribological grades modified with PTFE, molybdenum disulfide, etc.
- antistatic grades
- coloured grades
- grades with a lowered emission of formaldehyde for application in automotive industry in fitting elements of the passenger cabin of automobiles.

Meeting the demands of the market, the offer of construction plastics was broadened also by new grades of modified plastics: Tarnamid A based on polyamide 66 (PA66), Tarnoprop based on polypropylene copolymers and homopolymers (PPH and PPC) and Tarnodur A based on polybutylene terephthalate (PBT).

Table 3

Typical properties of polyamide 6 and polyoxymethylene grades at a temperature of 23°C

Property	Unit of measure	Tarnamid® T-27 NAT dry/ conditioned	Tarno-form® 300 NAT	Tarnamid® T-27 GF30 NAT	Tarno-form® 300 GF6 NAT
Glass fibre content	%	0	0	30	30
Melting point	°C	221	167	221	167
Density	g/ m³	1.13-1.14	1.41	1.35	1.63
Water absorption, max.	%	9.5	0.8	6.3	0.8
Moisture absorption, 50% RH		2.3-3.5	0.2	2.0	0.17
Yield stress during tension	MPa	78/45	62	-	-
Breaking stress	MPa	50	53	175/1110	125
Unit elongation during breaking	%	50/250	50	4/7	1.5
Elasticity module during tension	MPa	2800/1100	2800	9500/5700	10000
Notched impact resistance acc. to Charpy, 1eA	kJ/ m²	5/12	6	13/20	5
Impact resistance acc. to Charpy, 1eU	kJ/ m²	Does not break	180	90/100	25
Softening point acc. to Vicat, B50	°C	195/180	150	215/200	160
HDT, 1.8MPa	°C	60/50	115	200/195	160
Processing shrinkage	%	1.4/1.4	2.0	0.3/0.9	0.7/1.3

The offer of modified plastics was created as a result of intensive research and development works conducted at Zakłady Azotowe by the specialized Laboratory of Plastics. The R&D development with regard to plastics includes:

- designing new, modified grades of construction plastics
- perfecting the production technology of polymers
- testing the quality and safety of plastics and plastic products
- development of new applications of plastics and tests supporting new applications
- testing the processing properties of plastics
- pilot production of newly developed plastics grades
- developing and implementing new analytical methods.

The Laboratory of Plastics specializes mainly in testing construction plastics such as polyamides (PA) and polyacetals (POM), as well as other raw materials, i.e. granulates and products made from them.

Thanks to an experienced team of specialists and technical staff, modern and extensive instruments as well as many years of practice, it has the possibility to perform tests of plastics in a broad scope, i.e. physiochemical, mechanical, thermal properties, inflammability, ageing, processing, electric, rheological, colour and others. Moreover, it provides technological support to customers.

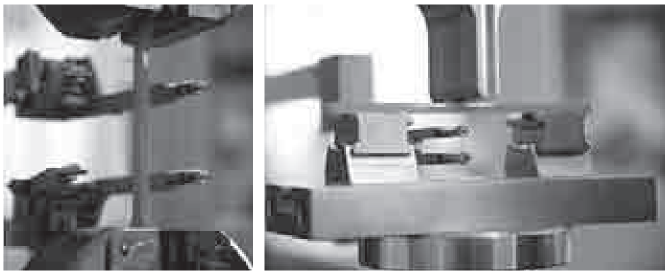


Photo 6. Tests at the Laboratory of Plastics of Azoty Tarnów

Azoty Tarnów Capital Group has adjusted the system of distributing construction plastics especially for customers. The company has implemented a system of direct sales. With the richest offer of construction plastics in the country, Azoty Tarnów guarantees attractive prices, quick realisation of supplies and post-sales service.

At the beginning of the 1990s, Zakłady Azotowe in Tarnów undertook efforts to implement quality management systems. In 1997 the first certificate was obtained from two independent certifying units – the Polish Centre of Research and Certification and Bureau Veritas Quality International – which confirmed the compliance of the implemented system with norm ISO 9002:1994. In the following years, the system was expanded by the scope of designing according to ISO 9001:1994, and then it was adjusted to the new norm ISO 9001:2000 and ISO 9001:2008. In 2002, a certificate of compliance was obtained with norm ISO 14001 for the Environmental Management System.

Currently, the integrated management system complaint with international standards includes:

- The Quality Management System compliant with norm ISO 9001:2008
- The Environmental Management System compliant with norm ISO 14001:2004
- The Food Safety Management System compliant with norm ISO 22000:2005
- Occupational Health and Safety Management System compliant with norms PN-N-18001:2004 and BS OHSAS 18001:2007
- The Responsible Care Management System compliant with the requirements of the European Chemical Industry Council.

The construction plastics offered by Azoty Tarnów Capital Group are subjected to certification by independent accredited units for compliance with specified requirements. The following certificates were obtained, among others:

Certificates: Tarnamid®:

1/ UL Certificate of Compliance for Tarnamid® T-27 NAT and Tarnamid® T-27 GF10-GF50, from 2011, so-called Yellow card,

2/ Certificate USP 26 for class VI plastics – National Institute of Public Health from 2004

Certificates: Tarnoform®:

1/ WRAS – Water Regulations Advisory Scheme 2011

2/ KTW issued by the Hygiene Instytut des Ruhrgebiets 2011

3/ Certificate of Drug Master File (DMF) 2010

4/ Certificate USP 26 for class VI plastics – National Institute of Public Health from 2003

5/ ACS issued by CARSO from 2008

Medals and awards:

The construction plastics offered by the Azoty Tarnów Group won a series of awards:

Tarnamid®:

1/ Prochemia 2004 Grand Prix: Tarnamid® antistatic and electroconductive

2/ Plastpol 2004 Medal: polyamide with decreased inflammability with low harmfulness of flame retardants

3/ Plastpol 2008 Award: Tarnamid® T-30 MHLS for stadium seats

4/ European Medal 2009: Tarnamid® T-30 MHLS

alphalon®:

1/ Plastpol 2011 Award: alphalon® E36LN

Tarnoform®:

1/ Plastpol 2005 Medal: Tarnoform® with antistatic and conductive properties

2/ Plastpol 2009 Award: Tarnoform® - low deposit
3/ Plastpol 2012 Medal: Tarnoform® with lowered emission of formaldehyde for automotive applications.

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Krzysztof PIEŃKOWSKI - M.Sc., is a graduate of the Faculty of Chemistry of the Cracow University of Technology. He specialises in the technology of manufacturing and processing plastics. He is co-author of several patents, publications, elaborations and implementations with regard to polyamide 6 and polyoxymethylene. For several terms he was a member of the Supervisory Board of Zakłady Azotowe in Tarnów-Mościce S.A. Currently, he is the Chairman of the Board at ATT Polymers GmbH in Guben, Germany which belongs to the Azoty Tarnów Capital Group.