Optical Organic Glass; Know-how to Polish Layers

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The study consists of a new know - how applied to the organic optical glass used to manufacture optical components. The study was developed on several organic optical glasses, usually used to manufacture pieces with optical properties. The innovative methods, subject of this paper, are manufacturing active optical surfaces with a high quality surfaces. That is the reason of complex control, based on different methods which can reveal changes of inner or outer structure thinking of nanometric level.

Keywords: organic optical glass, nanometric structure

The optical organic glass is used, mostly, for complex optical components and especially for those with aspheric surfaces or curves. A low specific density is a major advantage and the reason to be spread used in glasses technology.

Organic glass is a fully synthetic plastic material available in a vitreous state. It consists of macromolecular organic compounds which do not follow any principle of periodic arrangement and are hence amorphous.

In most cases, duromers are used to produce plastic lenses made of organic glass [4]. Once they have been thermally treated after production, their shape can no longer be changed.

A typical feature of the production process is that, when subjected to heat, many molecules (monomers) combine to form giant molecular chains (polymers) as a result of chemical reaction. The well-known plastic CR 39 is one of the organic materials used for plastic lenses [7].

Some advantage recommends using plastic materials:

·high refractive indices allow the production of thin lenses, even for higher prescriptions; range of refractive indices from n = 1.5 to n = 1.665;

·lightweight spectacles which are comfortable to wear; low density;

·very suitable for sports and children's spectacles; high resistance to breakage;

·tinting using dipping process, irrespective of prescription, in whatever color the wearer requires; extensive tinting possibilities [1].

The experiments have been focused on upper layers behaviour during mechanical and chemical action, using different types of polishing materials with micro and nano structure and texture. The components have been added to a liquid suspension along with another emulgators to make final product smooth and soft. The homogeneity of final product was another issue to be achieved, along with reducing bubbles and hardness of solution in time.

Table 1
COMPARISON BETWEEN CHARACTERISTICS OF POWDERS USED IN CLASSICAL POLISHING METHODS AND NANOPOWDERS

Type of powder/ technical characteristics	Micro powder for polishing	Nanopowders for polishing	
		Dimension <1μ	Dimension <0,5μ
The average dimension of particle	1,5-2μ	≤0,9μ	0,05-0,4μ
Chemical composition (base active substance)	CeO_2< 60 %	CeO_2> 99 %	CeO_2>99 % or Al_2O_3>99,99 %
Uniformity	Particle contain 15μ ≤ 0,1%	Particle contain 15μ ≤ 0,05%	-

Table 2
THE MAIN CHARACTERISTICS FOR POLISHING NANOPOWDERS

Type of nanopowder/ technical characteristics	AUROPOL 0048	CERI HPC	LINDE A	LINDE B
The average dimension of particle	0,4-0,6μ	0,5-0,9μ	0,3μ	0,05μ
Chemical composition (base active substance)	CeO_2> 99 %	CeO_2> 99,4 %	Al_2O_3> 99,99 %	Al_2O_3> 99,99 %
Color	White	Light brown	White	White
Cristal structure	cubic	cubic	hexagonal flat	hexagonal flat
pH in solution	3,7-4,8	6,5±1	-	-
Density (g/cm^3)	-	7,0-7,5	4,0	4,0
Solubility	No solubility in water	No solubility in water	No solubility in water	No solubility in water
Supplier	Auer Remy Germania	Teller Germania	Universal CO-SUA	Universal CO-SUA

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Table 3

TECHNICAL CHARACTERISTICS OF EMULSIONS READY TO USE

Type of nanopowder/ technical characteristics	CEROX 1650	ULTRA TRENT 1.1
The average dimension of particle	1,5-2μ	1-1,2μ
Chemical composition (base active substance)	CeO_2<60%	CeO_2 - 99,98 %
Color	white	white
Cristal structure	-	cubic
pH in solution	-	7-9
Density (g/cm^3)	1,5-2,0	7,132
Solubility	insoluble	insoluble
Supplier	Rhodia-Franta	Trent Mann Prod. LTD Anglia

Experimental part

Materials used for polishing; powders and emulsions

The main characteristics of this powders and emulsions for nanopolishing are the average dimensions and chemical structure [3]. Unless the micro powders for polishing used for optical surfaces, sorts were to be used in experimental steps of nanopolishing phase are highly chemical pure (active substances are over 99%), the average dimension of particle is around hundreds of nanometers and uniformity is very high.

Nanopolishing suspension is made with distilled water, powder concentration being: 200 g of powder in 1L of distilled water.

Materials used to support polishing emulsions

During experimental phases the support was mastic half hard made: 50% polishing mastic P65 NIOR 1158 and 50% polishing mastic P55 NIOR 1158.

Polishing technologies

The experiments have been carried out on mechanical specialized device, but powder supplying was manual [1].

Two different polishing technologies have been used:

a) classical way, one step of polishing and suspensions was very concentrated and the polisor was wet all the time;

b) combined way, consists in a classical way of polishing, during which, the polisor is kept wet with the liquid of polishing suspension, nanopowder beeing down in the solution.

Technological parameters of polishing device

Experiments took place on a Karger machine with 10 working places. Working parameters have been set up to the following values:

-samples block diameter: 60 mm;
-samples number in block: 7 pieces ;
-diameter polishing device: 80 mm;
-rotation speed of lower shaft: 100 rot/min;
-number of oscillations of upper shaft: 72 osc/min;
-working pressure, located in the center of block of samples: 0.5 kg/m^2.

Preparing of samples blocks

Both surfaces were sanded; the level of sanding depends on the position of surfaces linked with the checked surface. Both surfaces have been polished on Karger machine, to the point of leveling all the smallest holes which could be seen with a 6x magnifier. Then on the main surface polishing continue to the upper level fine and ultrafine [2]. Suspension supplying was manual by brushing, keeping the polisor wet all the time.

Evolution was in the attention all the time, and regularly, in the first 10 min and aftermath from 30 to 30 min, till the final result was achieved when one surface was free of any small holes, visible with a 6x magnifier.

The samples with finished surface have been taken away from the blocking device, manually washed with technical perchlorethilen and absolute ethylene alcohol and controlled for surface accuracy, for getting the eventual macro defects (scratches and points with dimensions over 0,01 mm, stains or unfinished zones). Finally, the roughness was determined by atomic force microscope.

Results and discussions

The conclusions of evolution process are synthetically presented in table 4.

Manufacturing under layers of organic optical glass, Perspex and Moden glas by suspensions based on powders with average particle dimensions around hundreds of nanometers (300-600 nm) will lead to a well polished surface pre-polishing being unnecessary [10].

Nanopolishing with suspensions based on nanopowders of polishing with average dimensions of particles hundreds of nanometers lead to less quality of surface [9]. The experiments was lead on layers of Moden glas prepolished with suspensions based on aluminum oxide with average dimensions of particles 300 nm – Linde A, Universal Co, USA, followed by polishing with suspensions based on aluminum oxide with average dimensions of particles 50 nm Linde B, Universal Co, USA.

Nanopolishing with particles of average dimensions of hundreds of nanometers was no longer in time then with particles with average dimensions of micrometers, the other parameters being the same; two keys technological factors lead to this [8]:

- layers had initial roughness smaller than conventional polished layers;
- polishing suspensions under micrometers used for experiments contain active substances (> 99%), comparing with polishing suspensions based on micrometers powders (60%) active substances;
- the lasting of polishing process depends on initial roughness of a layer (table 5).

It's easy to see that very fine polished surfaces, with one or 2 granulation, are faster polished than fine polished surfaces. There is no solid evidence that time or suspension concentration influences surface quality; the roughness of optical glass Perspex is the same no matter what concentration or time was (table 6).

With some exception (suspensions based on AUROPOL 0048 for Perspex optical glass), polishing has a fast debut, surface being finished or almost finished, after 10 min of work, even polishing regime was pretty slow.

Table 4

MANUFACTURING EVOLUTION ON SURFACE USING UNDER-MICROMETERS SUSPENSIONS

Polishing suspension	Material layer	Final polishing	Polishing type	The stage of surface manufacturing			Observations
				After 10 minutes	After 40 minutes	Completing polishing	
Cerox 1650	Perspex	W6	Classical	Clearing	Polishing 30%	180 min.	
Ultra trent 1.1	Perspex	WCA 5T	Classical	Clearing	Polishing 30%	90 min.	Samples lot P3
Auropol 0048	Perspex	WCA 5T	Classical	Not clearing	Polishing 30%	90 min.	Samples lot P1
CERI HPC	Perspex	WCA 3T	Classical	Partial clearing	Polishing 30%	90 min.	Samples lot P5
	Moden glass	WCA 3T	Mixture	Clearing	Polishing 70%	75 min.	Samples lot QP2
Linde A	Moden glass	WCA 3T	Mixture	Partial clearing	Polishing 20%	150 min.	Samples lot QPL
Linde B	Moden glass	Polished with Linde A	Classical	After 15 min. of polishing the surface is not good, a continuous stain layer appearing of oxide was observed			Re-polishing cu LINDE A
Platinum trent	Perspex	WCA 5T	Classical	Clearing	Polishing 30%	90 min.	Samples lot P2
	Moden glass	WCA 3T	Mixture	Clearing	Polishing 50%	120 min.	Samples lot QP1
CE 480 ST	Perspex	WCA 5T	Classical	Partial clearing	Polishing 20%	150 min.	Samples lot P4

Table 5

THE LASTING OF POLISHING PROCESS DEPENDS ON INITIAL ROUGHNESS OF A LAYER

Layer	Nanopowder	Polishing type	Initial roughness R_a	Polishing time	Percentage (%)
Perspex	CERI HPC	Simple	0,08µ	1,5 hours	100
Perspex	CERI HPC	Simple	0,3µ	2,0 hours	134
Perspex	CE 480 ST	Simple	0,2µ	2,5 hours	100
Perspex	CE 480 ST	Simple	0,3µ	3,0 hours	120

Table 6
THE INFLUENCE OF DURATION AND CONCENTRATION UPON NANOPOLISHED SURFACE QUALITY

Layer	Polishing nanopowder	Polishing type	Nanopowder concentration	Polishing time	Roughness RMS (nm)
Perspex	CERI HPC	Simple	20%	1,5 hours	1,376
Perspex	CERI HPC	Combined	20%	2,0 hours	1,282
			<1%	0,5 hours	

Table 7
SURFACE QUALITY NANOPOLISHED DURING EXPERIMENTS

Polishing suspension	Sample	Under layer material	The last stage of polishing	Polishing type	Manufacturing quality		
					Roughness RMS (nm)$^{1)}$		Clearness 5/ ISO 10110$^{2)}$
					22μ	44μ	
CEROX 1650	$^{3)}$	Perspex	W6	Classic with Automatic action	**-**	**8,87/ 7,398***	3x0,25
CEROX 1650	$^{3)}$	Perspex	W7	Classic with Manual action	**2,59**	**4,42**	3x0,16
ULTRA TRENT 1.1	P3-2	Perspex	WCA 5T	Classic	-	3,437	max. 2x0,10
AUROPOL 0048	P1-4	Perspex	WCA 5T	Classic	-	**1,63/ 1,716***	max. 3x0,16
CERI HPC	P5-1 P5-5	Perspex	WCA 3T	Classic	1,576	2,138	max. 1x0,10
	P5-2V	Perspex	WCA 3T	Combined	**1,282**	**-**	max. 1x0,10
	QP2-5	Moden glass	WCA 3T	Classic	-	3,115/3,187*	max. 2x0,10
PLATI-NUM TRENT	P2-2	Perspex	WCA 5T	Classic	-	2,084	max. 3x0,16
	QP1-4	Moden glass	WCA 3T	Classic	-	3,656	max. 2x0,10
CE 480 ST	P4-5	Perspex	WCA 5T	Classic	1,44	**-**	max. 2x0,10
	QP3-1	Moden glass	WCA 3T	Classic	-	3,321	1x0,10 C1x0,10

1) measured with atomic force microscope by direct contact;

2) seen following IT-DP 04 PRO OPTICA;

3) data achieved during project NETIMAGE;

* measured in the center and on the edge of sample;

The behaviour of nanopolished layer was different, in the same polishing conditions [1]: so, Moden glass, which has less hardness than Perspex, is harder to polish than Perspex with the same polishing suspensions, in which the average dimension of grain is 0.6 µm (Platinum trent, AUROPOL 0048, cerium oxide based and Linde A aluminum oxide based) but very easy to polish with CERI HPC cerium oxide based with average dimensions less than 0.6 µm and ready to use emulsion CE 480 ST with average dimension around 0.9 µm. Optical glass Perspex is polished in classical way in the same time for 3 out of 4 suspensions based on under nanometer powders of cerium oxide (CERI HPC, PLATINUM TRENT and AUROPOL 0048). The same glass is harder to be polished with CE 480 ST specially designed for soft glasses.

Measuring roughness, surface optical quality viewed by 10x magnifier and micrometer defects remained on surfaces have been done for surface characterization finished with under micrometers polishing method [5]. The results are presented in table 7.

Roughness for polished surfaces have been measured with atomic force microscope (AFM), NOMAD and Q-SCOPE brand by Quesant, USA, and the results are presented in table 8. Analyzing the results, some conclusions are very clear:

·RMS values of roughness are diminishing if the polishing suspensions are under micrometers powder base (CEROX 1650), comparing with those done by polishing with micrometers suspensions with bigger grounds;

·RMA values are diminishing if initial roughness is smaller;

·Optical glass Perspex under layer, polished with nano-particles of cerium oxide, gains RMS values around 2 nm/ 44 µm [6].

Figure 1a, nano-polished surface texture with CERI HPC on sample of Perspex. It's easy to see the traces of micro particles on surface as very fine digs:

·the smallest value of roughness RMS was achieved by nano-polishing using suspension based on aluminum oxide LINDE A;

Table 8

MEASURED DIMENSION OF NANO-DEFECTS UPON NANO-POLISHED SURFACES

Sample	Nano-polishing suspension	Highest/Longest (nm)	Deepest/Longest (nm)	Observation
Perspex	AUROPOL 0048	+5,28 / 853	-16,54/719,9	
Perspex	CERI HPC	+3,8/153;	-1,11/255,4	
Perspex	CERI HPC	+12,8/716,8;	-15,13/949,7	
Perspex	PLATINUM TRENT	+71,4/778,2	-	Probably clusters
Perspex	CE 480 ST	+6,43/747,1	-9,98/680,8	
Moden glass	LINDE A	+8,25/248,6	-13,43/348,1	Multiple nano-defects
Moden glass	PLATINUM TRENT	+70,81/855,9	-4,53/802,8	Probably clusters
Moden glass	CERI HPC	-	-71,06/1.170	
Moden glass	AUROPOL 0048	+18,72/629,2	-4,17/640,4	

- nano-polished surface texture, has prints of micro particles shaped as fine digs (fig. 1a & 1b), which means the over layer was not enough getaway.

Figure 1b, nano-polished surface texture using LINDE A on samples Moden glass. It's easy to see the prints of micro particles for polishing as very fine digs and lots of nanometers clusters.

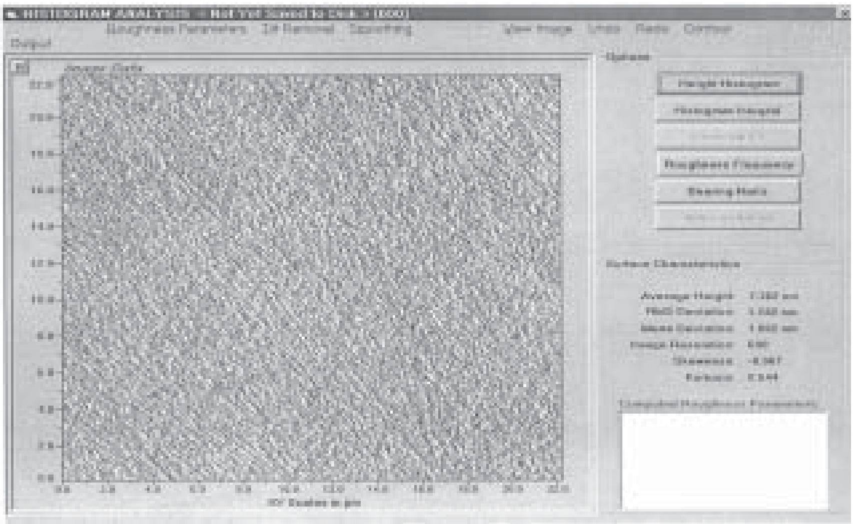

Fig. 1a. CERI HPC nanopolished surface texture for Perspex sample

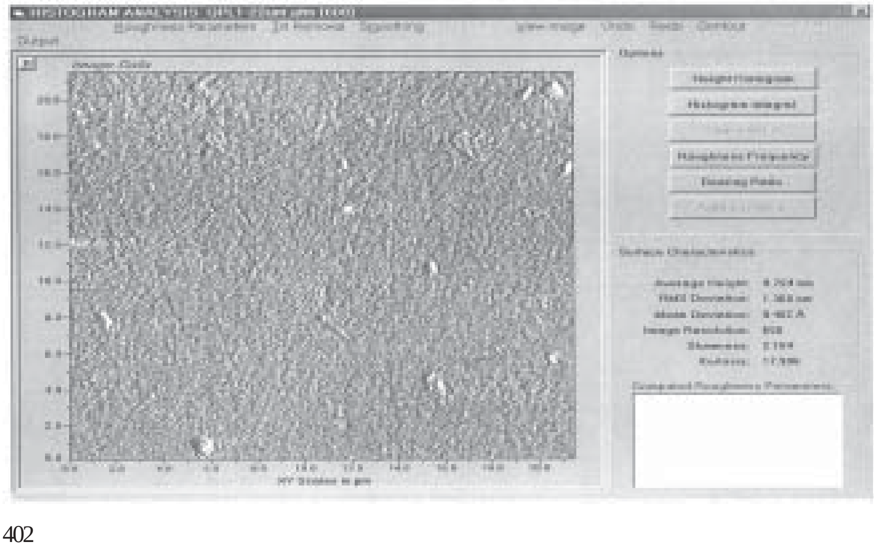

Fig. 1b. LINDE A nanopolished surface texture for Moden glass sample

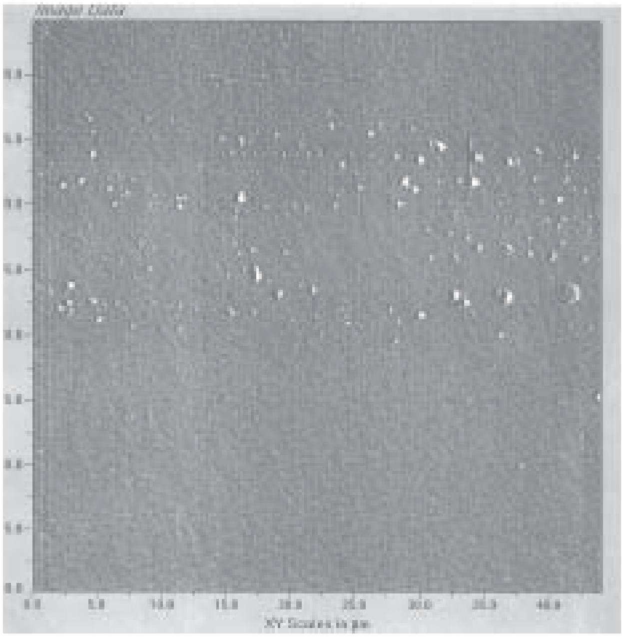

Fig. 2. AFM image for under-micrometers points on surface

Most of samples measured highlights lots of unusual patterns (fig. 1b, fig. 2) crowded or not, dimensions measured with atomic force microscope [5]. In table 8, it’s shown a synthesis of measures done on polished samples with different polishing suspensions. The scanned area is 44 . 44 μm^2.

Figure 2 – highlighting using AFM:

The presence of under-micrometers crowded particles are due to small pieces of polish materials or atmospheric particles.

Accuracy

After a carefully analyze of results presented in table 8, it’s easy to see that surfaces gained by nanometers powders polishing and very fine pre-polished have less macro-defects of accuracy according to standard SR ISO 10110-7, 5/nx0.1 grade.

Conclusions

Micrometers defects on nano-polished surface, with side/diameter less 0.01 mm, are linked to polishing level, as standard SR ISO 10110-7 set.

Polishing level checking is done by two methods:
- by checking glaze effect on polished surface;
- by binocular microscope LEITZ brand;

Checking glaze effect on polished surface

This method is based on global checking of polished surfaces finishing and consists in carefully observation of glaze on checked surface, glaze can be continuous or discrete. The assessment is based on human abilities, experience and skills. This test is used in optical industries, like a quick test for polished surfaces with special conditions. The observation is done by setting the surface against a very power light source and gentle sloping the surface under different angles till glaze appears or not. The test was applied on seven samples of PERSPEX polished on both surfaces with combined polishing method with CERI HPC. The checked surfaces are fewer glazes.

Binocular microscope LEITZ method

Binocular microscope LEITZ used was DIALUX 20 EB model with 40x objective and 10x eyepieces.

Checking was done on a few samples polished with CERI HPC. The whole image field was observed several times on 10 mm long surface and every distinctive point was take into account and marked. Afterwards their dimensions were measured in depth with LEITZ microscope and in length with ZKM 01-250C Zeiss brand.

Nanopolished surface checked have limited number of micro-defects of accuracy (pitches of holes less 0.01 mm). That’s why, nanopolished surfaces can be considered P_3 polishing grade, because have less 16 points of interest on the whole surface.

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