

Applying the Sol–Gel Method to the Deposition of Nanocoats on Textiles to Improve Their Abrasion Resistance

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ABSTRACT: The thin-coat finishing of textiles carried out by the sol–gel methods gain greater and greater importance owing to their suitability for the versatile functionalization of textiles to impart to them properties being difficult and even impossible to obtain with the use of conventional finishing methods. This article presents the test results of the thin-coat protective finishing of cotton fabrics against abrasion. This treatment consists in depositing hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols synthesized from two precursors: (3-glycidoxypropyl)trimethoxy-silane and

aluminum isopropoxide on fiber/fabric surface. The abrasion resistance of the fabric treated by the sol–gel method has been increased by about five times according to Martindale test and this effect is resistant to prolonged laundering. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 3058–3067, 2012

Key words: textile woven fabric; cotton fabric; sol–gel coating; thin-coat finishing; hybride modified sol; xerogel; performance durability of textiles; abrasion resistance

INTRODUCTION

One of the most effective ways of increasing the service durability of textiles is the application of protective coats on the surface of fibers. Such coats are characterized by a high hardness and resistance to abrasion that protect the generally soft and sensitive to abrasive forces fiber material against external mechanical effects. This is particularly important in the case of the most sensitive natural fibers such as cotton and slightly less important in the case of more resistant synthetic fibers such as polyester or polyamide fibers. In this respect, great expectations are associated with the so-called nanocoating finishes

applied by the “sol–gel” technique. This technique is based on colloidal suspensions—sols—made from appropriately selected precursors, mostly metal oxides or organometallic compounds, with metal or semi-metal alkoxides being most frequently used. The sols obtained in this way are applied on fiber/fabric followed by thermal treatments at elevated temperatures (drying and polycondensation) to obtain cross-linked lyogels containing a considerable content of liquid phase. The liquid phase is removed by drying and the fiber surface is covered with a thin porous layer. Further treatment at elevated temperatures, due to progressing polycondensation converts this layer into a cross-linked gel coat physically and possibly chemically combined with the fiber surface. The mechanical properties of this coat depend on the intensity of thermal treatment, i.e., temperature and duration. It has been found that under thermal conditions admissible for textile fabrics, i.e., such that cause no damage as a result of the thermal destruction of the polymer, the coat is developed to reach a cross-linking degree typical for xerogels. Such products, showing a lower hardness and mechanical resistance than those of ceramic materials requiring a thermal treatment at about 600°C, show however a considerably higher elasticity, which is of primary importance in textile applications. Through a proper selection of precursors

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and conditions of sol synthesis as well as doping the sols obtained with appropriately selected functional nanoparticles, e.g. Al_2O_3 , and conditions of sol application on textile fabrics including further thermal treatments (drying, heating/cross-linking) one can obtain hard protective coats on the fiber surface imparting an increased abrasion resistance to the finished fabrics.¹⁻¹³

However in reality, the issue of anti-abrasive coating by the sol-gel technique is not so simple. Firstly, the applied coats should not significantly change the "fibrous" character of fabrics, i.e. their natural soft and pleasant handle and good drapability. These should not excessively increase fabric stiffness and deteriorate the hygienic and aesthetic properties of fabrics. In other words, the xerogel coating applied should be as thin as possible, elastic, and transparent. Moreover, to provide a durable finish, these coats should be strong and durably combined with the fiber surface not only physically but also by chemical bonds. It should be mentioned that the coat thickness and its elasticity as well as its character and the degree of combination with the textile substrate are of primary importance for its resistance to fracture and chipping. Such a chipping taking place during the use and care of clothing made of fabrics coated in this way results in the formation of lots of fine hard particles with sharp edges that act on the fiber matter as abrasive dust and significantly accelerate the damage of textiles. Consequently, such a protective coat instead of improving can deteriorate the abrasion resistance of the textiles finished and accelerate their wear compared to analogous fabrics with no coat-finishing. Hence it is required to provide as low "sol-gel" coat thickness as possible, most often within the range of 150–500 nm.^{1,2,14} Therefore, although it is not terminologically justified to name such coats as nanocoats (100 nm is accepted as the limiting nano-value) such a name is commonly used in published reports. It should be stressed that the elasticity of coats directly depends on their thickness—the thinner the coat is, the higher its elasticity and physical adhesion force of combining with the fiber surface. According to various literature reports, such coats can be obtained by using sols with a concentration not exceeding 50 g/dm³. These sols, as colloidal suspensions in alcohol, alcohol-water, or aqueous medium, should not contain particles with dimensions exceeding 50 nm due to limited stability. This concerns both sol particles and various modifying doping nanoparticles, e.g. such as nanoparticles of Al_2O_3 , incorporated into the cross-linked xerogel coats to increase their resistance to abrasion.^{1,2,9}

As far as the force of coat combination with the fiber surface is concerned, numerous literature reports suggest that this force can be considerably increased by using properly selected organosilicon

precursors for the synthesis of sols. This makes it possible to synthesize sols containing particles with functional groups capable of reacting and forming chemical bonds with the functional groups occurring on the fiber surface, mostly -OH groups (in the case of cotton).¹⁵⁻¹⁷

Good results can be obtained by the modification of pure silica sols with the addition of epoxysilanes such as (3-glycidoxypropyl)trimethoxysilane (GPTMS). Such an additive increases the adhesion of nanosol coat to the fiber surface, and consequently increases the fabric mechanical resistance. The improvement in adhesion is due to the development of chemical bonds between the "sol-gel" nanocoat formed on the fiber surface and the functional groups of fibers. Under the conditions of sol synthesis and following thermal processes, the epoxy ring of GPTMS opens and reacts with the hydroxyl groups on cotton surface. From literature reports it follows that the addition of GPTMS also increases the hardness of the inorganic-organic hybrid coat and improves the degree of combination between the "sol-gel" coat and the cotton fiber surface. The addition of GPTMS also exerts a beneficial effect on the elasticity of the coats obtained. Thus one can state that the contribution of this compound to the sol synthesis allows one to expect an increased resistance of such coats to chipping and consequently improved service durability (wear resistance) of the coated textiles.¹⁵⁻¹⁷ The elasticity of the "sol-gel" coats can be also increased by the modification of appropriate organic sols with cross-linking agents or softeners. However, increased quantities of organic materials will most likely adversely affect the abrasion resistance of the coats formed.^{1,2} For examples, this can concern the addition of poly(vinyl alcohol) used. The stiffness of textiles finished with nanocoats depends not only on the coat thickness but also on the so-called bridging or gel combinations between adjacent fibers. Such bridging stabilizes the mutual position of adjacent fibers and does not allow their displacement, which obviously decreases the elasticity of the textiles under finishing. The bridging takes place especially in the case of applying sols with too high concentrations or partly gelled and applied in excessive quantities. Such conditions also cause a considerable increase in the coat thickness.

A very important problem is also the durability of the increased abrasion resistance of textiles under conditions of prolonged use and care, which is directly connected with the coat adhesion to the fiber surface and the chemical bonds formed.^{18,19} Presenting the significant positive influence of the low "sol-gel" coat thickness, the abrasion resistance of a coat directly depends on its thickness. In the case of thin "sol-gel" coats, their abrasion or scratch resistance is a function of the thickness of the "sol-gel" protective

layer. For specified fabrics it can be assumed that the thinner the coat is, the lower its resistance to external damaging mechanical effects.^{1,2,9,18,20}

Based on the literature review and our own studies, we can conclude that this dependence is of primary importance for the “sol-gel” nanocoat finishing as it creates a discrepancy between the basic aim of the coating—expected increase in the abrasion resistance of fabrics—and the necessity of eliminating the disqualifying coat chipping and maintaining the “fibrous” character of textile fabrics under finishing.

Hence the apparently simple technology of the protective “sol-gel” coating against abrasion turns out to be problematic in practice. Although the same technology has been long used to harden plastic lenses or compact discs and to protect them against scratching,^{1,19} the “sol-gel” coating of textile fabrics to protect them against abrasion and to prolong their service life cannot be admitted as a completely mastered technology of industrial significance despite many positive reports by various research centers; it still remains a subject of research and tests.^{20–23}

EXPERIMENTAL

Materials

Textile fabrics

A commercial 100% cotton woven fabric with a surface weight of 150 g/m², a thickness of 0.36 mm, and plain weave (warp: 26 tex, 30 threads/cm, weft: 28 tex, 21 threads/cm) was used in this study. Samples of this woven fabric commercially finished, without any auxiliary agents (precleaning/desizing, bleaching, washing, and drying) were first additionally washed under standard conditions according to PN-EN ISO 6330:2002: “Textiles. Domestic Washing and Drying Procedure for Textile Testing. Procedure 5A” at a temperature of 40°C for 30 min., and then dried in a drier at 50°C.

Precursors

- Tetraethoxysilane 99.9% (TEOS) (Alfa Aesar GmbH & Co KG, Karlsruhe, Germany)
- GPTMS 98% (ABCR GmbH & Co KG, Karlsruhe, Germany)
- Aluminium (III) isopropoxide Al(OCH(CH₃)₂)₃ (ALIPO) (ABCR GmbH & Co KG, Karlsruhe, Germany)

Synthesis of sols

- (a) *Preparation of a “pure” silica sol with the use of tetraethoxysilane (TEOS)—Si(OC₂H₅)₄*

Silica sol was prepared by the hydrolysis of TEOS in an acid–water medium at pH = 3 on stirring with a high-speed stirrer for about 2 h at a temperature of about 80°C to obtain a transparent colloidal solution.

- (b) *Preparation of Al₂O₃ sol with the use of aluminum isopropoxide (ALIPO)—Al(OCH(CH₃)₂)₃*

Al₂O₃ sol was prepared according to the procedure given in the literature⁹ by mixing the ALIPO precursor with the mixture of ethanol and water (in a molar ratio of 2.5 : 1) using a high-speed stirrer for about 2 h at a temperature of about 80°C (boiling point of EtOH). The ALIPO/water molar ratio was 1 : 1. After that time, the pH value of the mixture was set at 3 by means of hydrochloric acid followed by the addition of poly(vinyl alcohol) (POCH S.A.). The stirring was continued until a homogeneous transparent colloidal solution was obtained.

- (c) *Preparation of a hybrid SiO₂/Al₂O₃ sol²⁴*

This sol was prepared by combining sols (a) and (b) in 90 : 1 volumetric proportion by an intensive stirring at 20–25°C.

- (d) *Preparation of a modified silica sol with the use of GPTMS precursor (SiO₂*)*

The silica sol modified with glycidoxo groups (SiO₂*) was prepared by the hydrolysis of GPTMS as in the case of the pure SiO₂ sol but in a neutral medium.

- (e) *Preparation of a hybrid SiO₂*/Al₂O₃ sol²⁴*

This sol was prepared by combining sols (b) and (d) in different volumetric proportions by intensive stirring at 20–25°C. In this way stable transparent sols were obtained.

All the sols were used in a concentrated state and/or after dilution with water in proportions of 1 : 10–40.

Preparation of sol-gel coats

The film-forming sols containing functional nanoparticles were applied on the fabric surface by padding with the use of a laboratory two-roller padding machine with a horizontal position of the squeeze rollers from BENZ GmbH (Switzerland), using the following padding conditions:

- Pressure: 1.47 MPa
- Padding rate: 1 m/min
- Squeeze out degree: 95–110%

The padded fabric samples were dried at a temperature of 60°C and then heated at 160°C for 1 min.²⁴

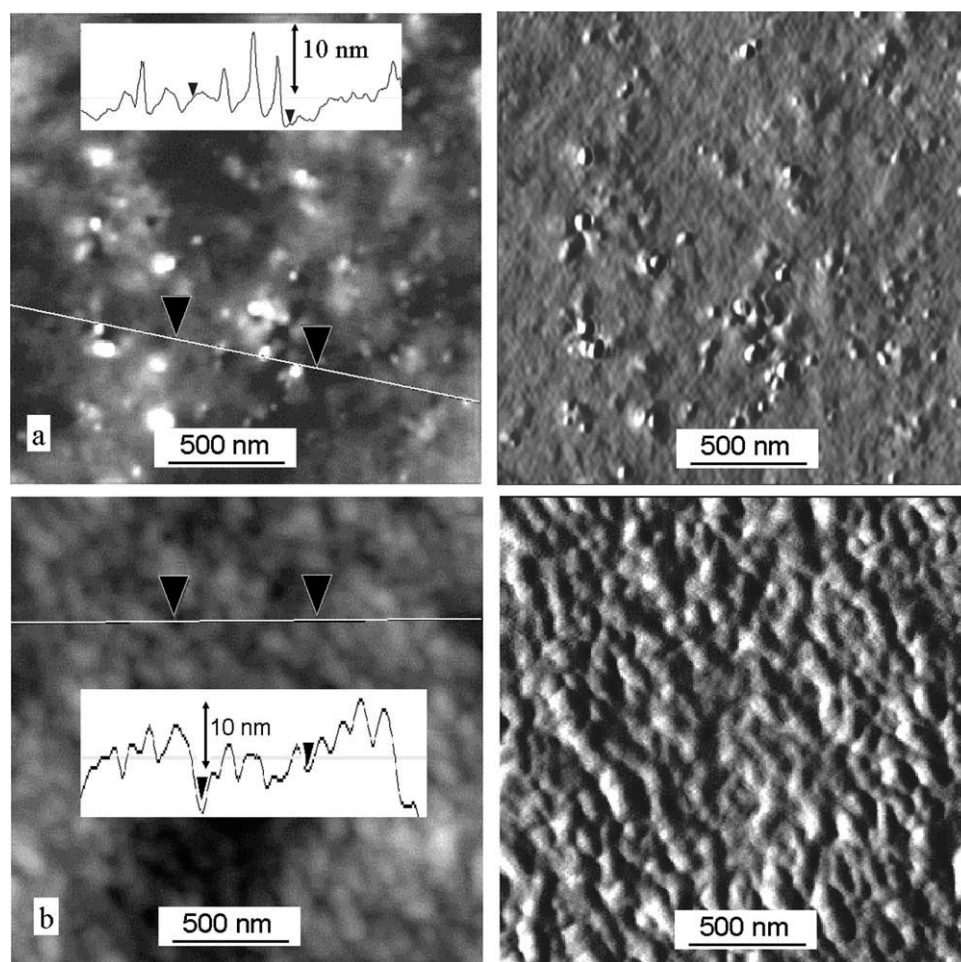


Figure 1 AFM images (left—height image, right—phase image) of thin layers on glass plates: (a) $\text{SiO}_2/\text{Al}_2\text{O}_3$ xerogel and (b) $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ xerogel.

Examination of the fiber surface with deposited sol-gel coatings

Microscopic observation of the fiber surface by means of SEM and AFM techniques

Scanning electron microscopy examination. The structure and morphology of the fiber surface with the xerogel coat deposited were examined by means of a JSM-5500 LV scanning electron microscopy (SEM, Jeol, Japan).

Atomic force microscopy examination. The structure and morphology of the fiber surface with the xerogel coats deposited and their changes due to wearing were examined by means of a Nanoscope IIIa atomic force microscopy (AFM) microscope from Digital Instruments (Santa Barbara, USA), using contact and oscillating modes.

Testing the resistance of fabrics finished with nanocoatings to abrasion

The tests were carried out according to PN-EN-ISO 12947-1:2000+AC:2006 "Textiles. Determination of the abrasion resistance of flat fabrics by the Martindale

method. Determination of sample failure²³ with the use of a Nu-Martindale 864 apparatus (James H.Heal & Co. Ltd., England). The pressure of abrasive heads: 12 kPa. A standard wool woven fabric was used as abrasive element. According to the standard, the test is terminated when two threads in the fabric tested are broken due to abrasion. At the same time there is recorded the number of revolutions of the abrasive head, after which the fabric was scraped out.

Testing the resistance of fabrics finished with nanocoatings to laundering

Abrasion tests were also performed for fabrics after five-fold laundering cycles performed according to PN-EN ISO 6330:2002. "Textiles. Domestic Washing and Drying Procedure for Textile Testing. Procedure 5A (40°C),²⁵ using a drum washing machine FOM 71MP LAB VASCATOR (Electrolux, Sweden).

RESULTS AND DISCUSSION

In the initial phase of the study, several series of coats, prepared with the use of SiO_2 and Al_2O_3 sols,

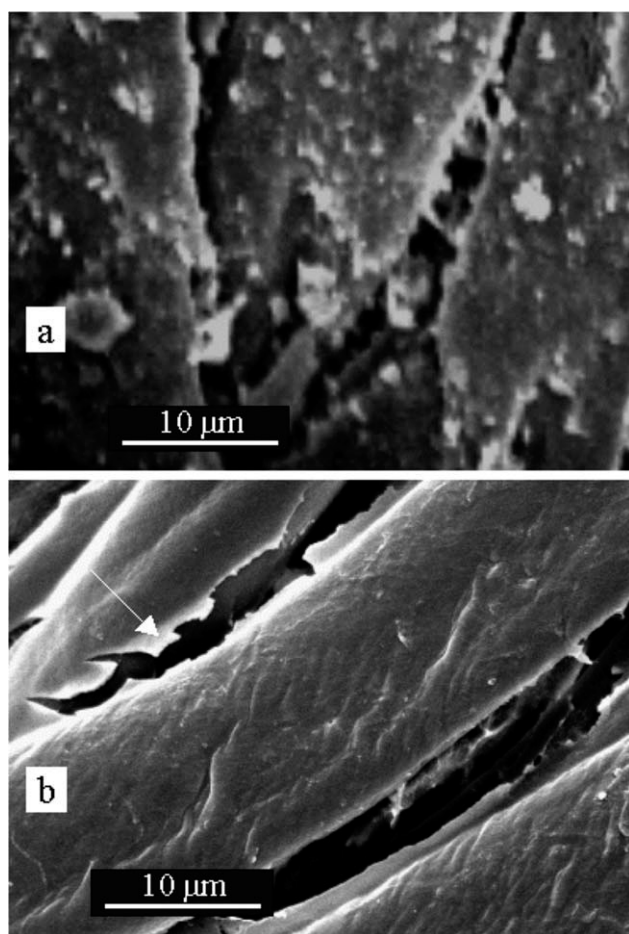


Figure 2 SEM images of fibers in the fabrics with deposited $\text{SiO}_2/\text{Al}_2\text{O}_3$ coat after 3000 cycles of abrasion: (a) visible chipped out coat particle; (b) visible cracks in the non-elastic coat between fibers (visible already before abrasion).

were deposited on the cotton fabric under investigation. However these xerogel coats appeared to be irregular, non-elastic, and brittle. They cracked already during lyogel drying and continued to crack in a subsequent heating operation. These drawbacks of SiO_2 and Al_2O_3 sols with respect to application, causing their rapid coagulation resulting in thick and rigid crackable coats, prompt us to synthesize hybrid $\text{SiO}_2/\text{Al}_2\text{O}_3$ sols by combining SiO_2 and Al_2O_3 on an intensive stirring for about 40 min. These sols were deposited on the cotton fabric, dried at 60°C and heated (cross-linked) at 160°C for 1 min to obtain hybrid xerogel coats.²⁴ As it was difficult to observe the coats deposited on the developed cotton fiber surface, these coats with a thickness of about $15\ \mu\text{m}$ were prepared on a smooth glass plate for further observations. AFM examinations clearly revealed the nanoparticles of Al_2O_3 with a size of about $15\ \text{nm}$, incorporated and uniformly distributed in the silica matrix [Fig. 1(a)]. The morphology of these hybrid $\text{SiO}_2/\text{Al}_2\text{O}_3$ xerogel was examined using SEM and AFM techniques. The elasticity of

the coats and their susceptibility to chipping were observed using SEM and AFM techniques. The abrasion resistance of the fabrics with deposited xerogel coats was determined by means of Martindale test.²³

It was assumed that the measure of the hybrid xerogel coat was its resistance to chipping under conditions of its intensive deformation by the abrasive head in Martindale test.

The results of testing the elasticity of the coats obtained appeared to be unsatisfactory, especially in the case of using concentrated $\text{SiO}_2/\text{Al}_2\text{O}_3$ sol. Already after a short time of the abrasive head action (after about 3000 cycles in Martindale test) one could observe coat chipping and bare fragments of fiber surface SEM image (Fig. 2) shows both these exposed fiber surface fragments and numerous chipped out fine coat particles with irregular size and sharp edges. The chipping of these particles undoubtedly results from the non-elastic xerogel coat. This feature has been already shown in the case of fabric before being subjected to Martindale test. As shown by the unsatisfactory abrasion test results [Fig. 3(a,b)], the chipped out particles of the xerogel coat act as abrasive powder. Therefore the coats of hybrid $\text{SiO}_2/\text{Al}_2\text{O}_3$ sol not only failed to effectively protect fibers but on the contrary, they decreased the abrasion resistance of the fabrics finished in that way, even below the values for untreated reference fabrics. This situation has been significantly changed when the sol in question was diluted with water, ethanol, or water-ethanol mixture (1 : 1). Then the abrasion resistance of cotton fabric was at a level of 30,000 cycles. In relation to the abrasion resistance of untreated fabric (20,000 cycle), this result indicates an increase by 50%. The results obtained are illustrated in Figure 3(a,c,d). These results indicate that the thickness of the coat

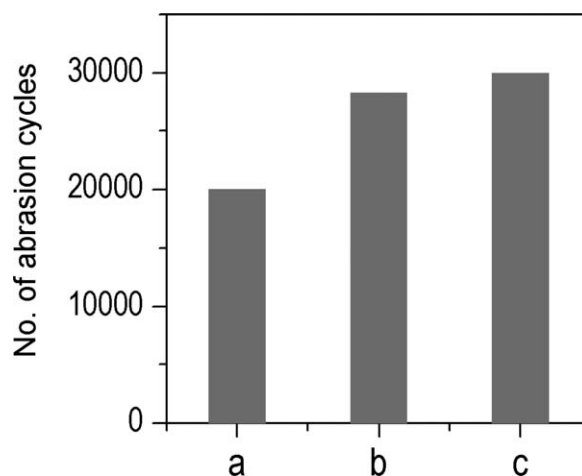


Figure 3 Abrasion resistance of cotton fabrics according to Martindale test: (a) untreated fabric, (b) fabric coated with the water-diluted sol [1 : 10], and (c) diluted with ethanol [1 : 10].

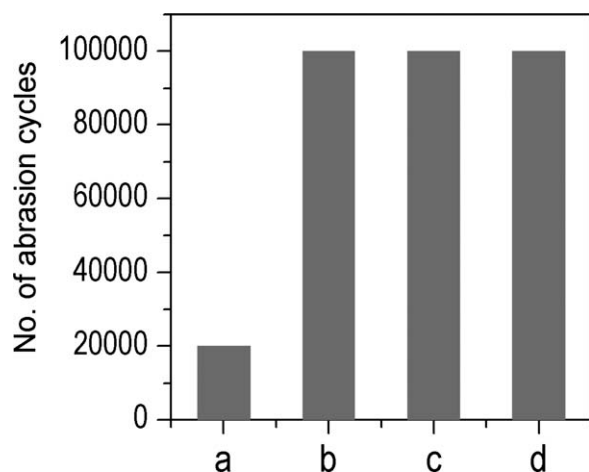


Figure 4 Abrasion resistance of cotton fabric according to Martindale test: (a) untreated fabric, (b) fabric coated with $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sol, (c) fabric as in (b) after a single laundering, (d) fabric as in (b) after fivefold laundering under standard conditions.

deposited exerts a considerable influence on its elasticity and the degree of its combination with fiber. Based on these results, one can also conclude that the type of solvent used practically exerts no effect on the abrasion resistance of xerogel coats and consequently on their elasticity and, most likely, structure.

Based on the test results obtained and the literature reports presenting the beneficial effect of the addition of GPTMS to TEOS in the synthesis of SiO_2 sol,^{15–17} assumptions for the synthesis of a hybrid sol with the use of two precursors: GPTMS and ALIPO were worked out and a method for its preparation was developed.²⁴ It was assumed that such a hybrid sol should make it possible to form xerogel

coats of a great hardness and abrasion resistance simultaneously showing elasticity and resistance to chipping. One may also expect that such coats will show a high adhesion to fiber surface, considerably assisted by a possible formation of chemical bonds. According to many reports,^{15–17} such bonds can be formed between the unhydrolyzable functional glycidoxy groups resulting from the GPTMS epoxy ring cleavage under the conditions of sol synthesis and the hydroxyl groups present in the surface layer of cotton fibers. Based on the assumptions developed, a series of hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols was synthesized obtaining clear transparent sols with a good stability.²⁴ To identify the structure of such xerogel coats, they were deposited on a smooth glass plate and observed by AFM. The observations performed revealed the presence of numerous Al_2O_3 particles with a size of about 15 nm thus being more uniformly distributed than those in the $\text{SiO}_2/\text{Al}_2\text{O}_3$ coat. They formed a continuous coat on the plate surface [Fig. 1(b)].

Hybrid GPTMS-modified $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sol, both in the concentrated and diluted (1 : 10) versions, was deposited on cotton fabric under specified conditions and then dried at 60°C followed by heating at 160°C for 1 min.²⁴ It was accepted that the use of such a high cross-linking temperature that does not cause yet thermal damages to fibers, would increase the cross-link density of the coat and so its hardness and abrasion resistance.

Samples of fabrics with deposited hybrid modified xerogels coats were examined using SEM and AFM techniques and subjected to the abrasion resistance Martindale test.

The abrasion resistance tests performed showed a very good protective action of the $\text{SiO}_2^*/\text{Al}_2\text{O}_3$

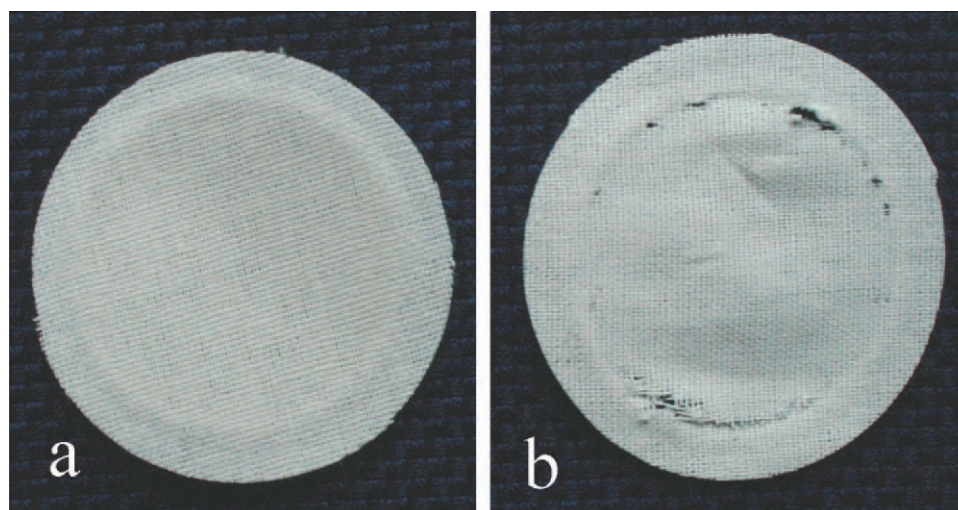


Figure 5 Photos of cotton fabric surface: (a) untreated fabric—abrasion after 20,000 cycles, (b) fabric coated with $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ —no visible damage after 100,000 cycles. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

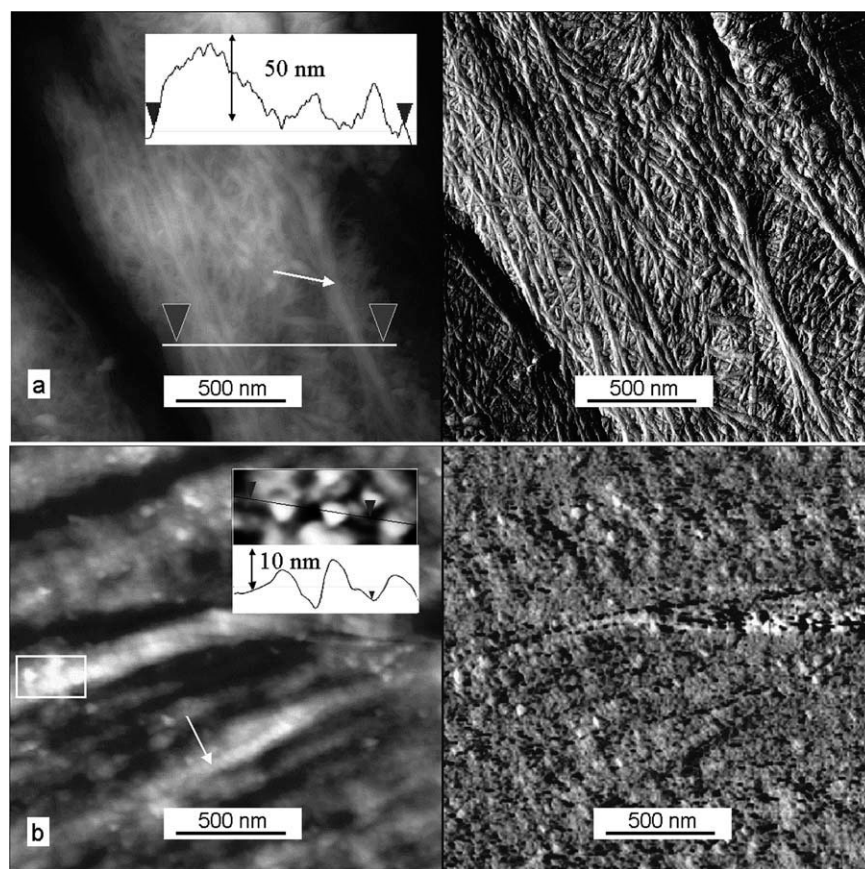


Figure 6 AFM images (left—height image, right—phase image) of the surface of fibers: (a) in untreated cotton fabric, (b) in fabric coated with $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ xerogel. Arrows indicate bundles of microfibrils (in (b) covered by the coat).

xerogel coats. The results obtained are shown in Figure 4. The nanocoat finishing of cotton fabric with the use of the hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols made it possible to obtain a perfect protection of fiber against abrasion exceeding 100,000 cycles of the abrasive head motion in Martindale test. Figure 5(a)

shows the untreated reference fabric that underwent abrasion already after 20,000 cycles and Figure 5(b) illustrates the fabric with deposited $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sol showing no visible signs of damage even after 100,000 cycles.

In order to determine the resistance of the coat finishing obtained to the conditions of maintenance and care, fabrics samples with deposited hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols were subjected to five-fold standard laundering at 40°C for 30 min²⁵ and then tested for abrasion resistance²³ and observed under a microscope. Test results presented in Figure 4 confirm the perfect abrasion resistance of the treated cotton fabrics, exceeding 100,000 cycles, both before and after their laundering. These results prove that the coat of hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ ²⁴ is very resistant to abrasion and prolonged laundering owing to its strong adhesion to fiber surface, probably through chemical bonds between the coat and the cotton fiber surface.

From the microscopic observations of the fabrics finished with hybrid modified $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols it follows that these sols are deposited on fiber surface in the form of elastic thin xerogel coatings that completely and uniformly cover the nanostructure of cotton fibers surface as is clearly shown by AFM

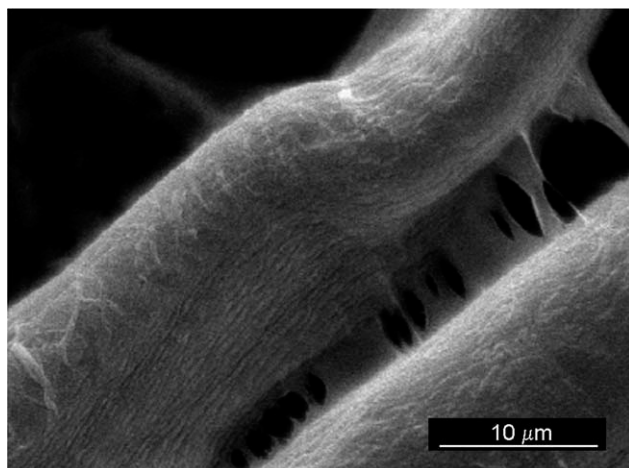


Figure 7 SEM image of fibers in the fabric coated with $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ xerogel (before abrasion test) —visible bridging between fibers formed by the elastic xerogel coat.

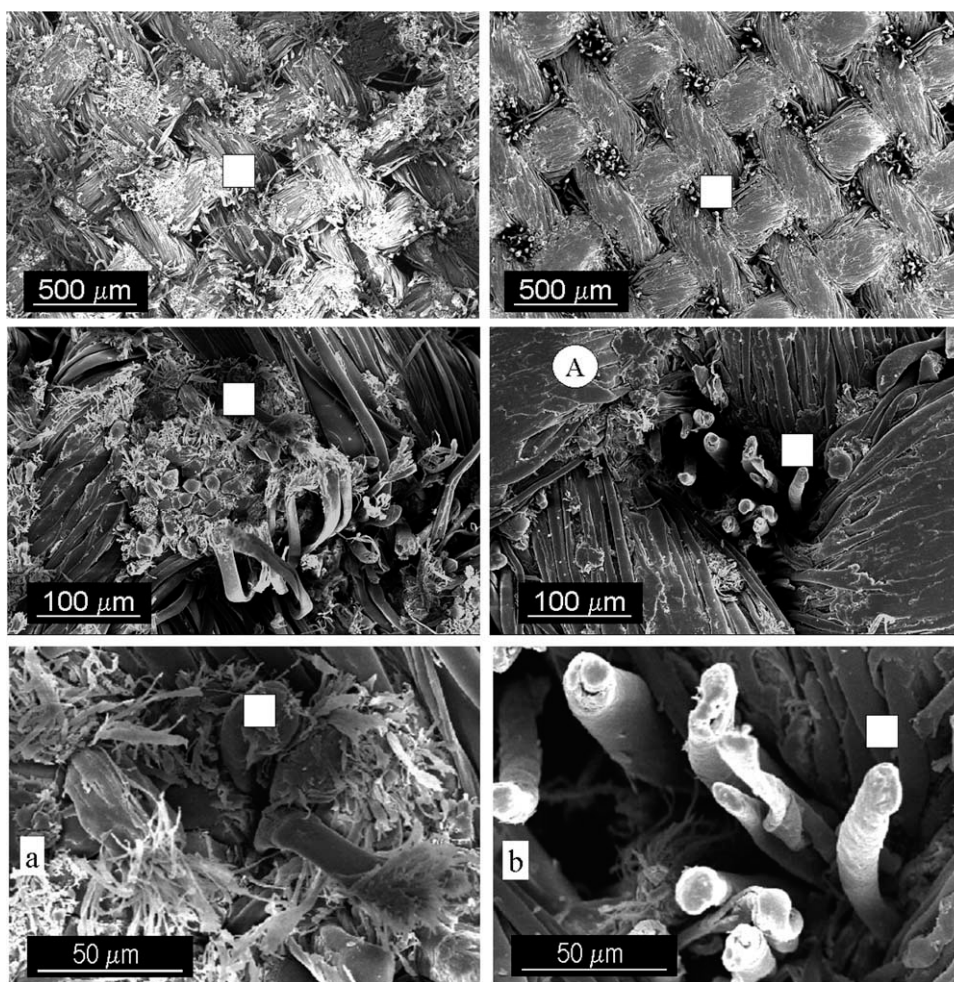


Figure 8 SEM images (various magnification) of cotton fabric after abrasion test: (a) untreated fabric—abrasion resistance 20,000 cycles; (b)—fabric coated with $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ xerogel after 100,000 cycles of abrasion. Area “A” shows a good condition of the coats deformed after abrasion.

image in Figure 6. As one can see in Figure 6(b), elementary fibrils [clearly seen on fibers in pristine fabric Fig. 6(a)] are uniformly covered by the coat. The rows in Figure 6(b) (indicated by arrows) reveal thicker bundles of fibrils covered by the coat. The bundles of fibrils protrude by about 20–30 nm. We therefore assume that the thickness of the coat is of the order of several tens of nm.

The difference in the elasticity of coatings made from both types of hybrid sols, i.e. $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{SiO}_2^*/\text{Al}_2\text{O}_3$, is shown by SEM images in Figures 2(b) and (7), respectively. These images prove that hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols show good film-forming properties and form elastic coats whose stretched fragments are seen between fibers (with no cracks) (Fig. 8). Owing to this great coat elasticity, fibers can be relatively displaced, although to a limited extent, and positively influence their abrasion resistance as well as tensile strength. Contrary to this, in the case of $\text{SiO}_2/\text{Al}_2\text{O}_3$ coats considerably more advanced interfiber links in the form of non-elastic bridges can

be observed. The cracks visible in Figure 2(b) result from their brittleness. There is also visible a great irregularity of these coats compared to the uniform thin coatings obtained from $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols.

SEM images also present great differences between the condition of $\text{SiO}_2/\text{Al}_2\text{O}_3$ coats and that of $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ coats on fabrics after abrasion resistance tests. If in the first case, one can observe considerable damage to fibers and coats including crumbled coat particles (Fig. 2), in the second case, the protective xerogel coats remained intact even after the prolonged action of the abrasive head (with wool abradant) showing only some flatness (Fig. 8). These images indicate completely different characteristics of both types of coats and equally different fiber protection.

The protective action and strong combination of the $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sol coats are well illustrated by SEM images shown in Figure 9. In the case of untreated fabrics, the action of abrasive head results in a clear fibrillation of fiber ends [Fig. 9(a)], while

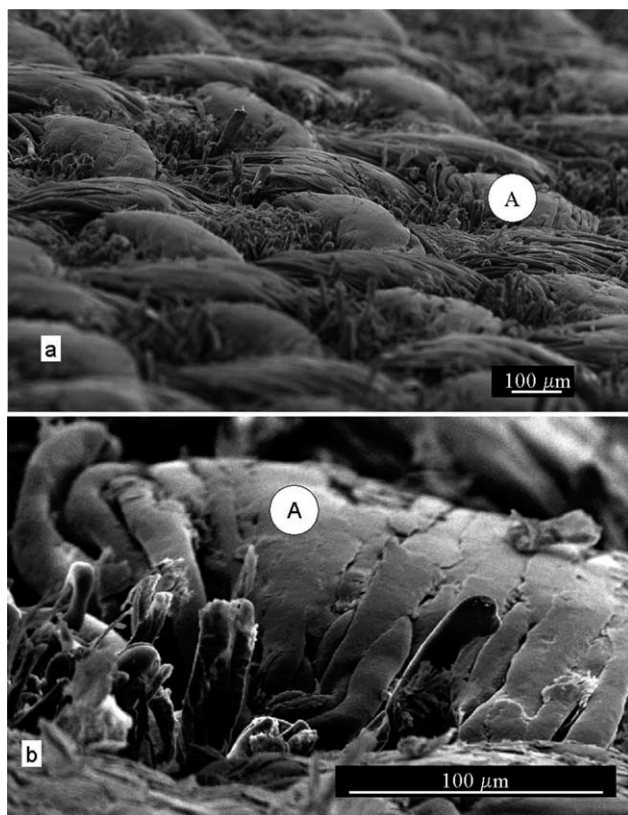


Figure 9 SEM images, taken at an angle of 10° , of cotton fabric coated with $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sol after abrasion tests. Area "A" shows a good condition of the coats after abrasion.

in the fabrics coated with the modified $\text{SiO}_2/\text{Al}_2\text{O}_3$ xerogel, individual fibers are enclosed by the durable coat protecting them against fibrillation [Fig. 9(b)] and the fiber ends remain undamaged with no signs of fibrillation.

The synthesis of hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols and the protective coats containing these sols deposited on cotton fiber/fabrics are the subject of patent application.²⁴

CONCLUSIONS

1. The test results presented confirm the soundness of assumptions worked out for both the synthesis of hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sols and the conditions of their deposition on cotton fiber surface in the form of xerogel coatings.
2. The deposition of hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$, xerogel coats on cotton fiber/fabric surface provides a very good and durable protection of these fibers against abrasion under both the use and care conditions, which constitutes a significant increase in the service durability of the cotton fabric finished with those coats. The abrasion resistance values exceed 100,000 cycles in the

Martindale abrasion test, which indicates a fivefold increase in abrasion resistance compared to that of untreated fabrics. A similar high abrasion resistance of the treated fabrics is maintained after a prolonged laundering under standard conditions.

3. The high wear resistance of the hybrid xerogel coats deposited on fiber surface under the use and care conditions determined by the Martindale abrasion test finds its confirmation in the outcomes of SEM and AFM examinations. The hybrid xerogel coats uniformly and continuously cover the fiber surface and are characterized by a high elasticity and resistance to chipping and a good combination with fiber surface.
4. From the SEM and AFM images of fabric samples with deposited hybrid $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ xerogel coats it follows that these two types of sols show different characteristics and mechanisms of abrasion/destruction of the coatings obtained.
5. The developed synthesis of hybrid $\text{SiO}_2^*/\text{Al}_2\text{O}_3$ sol and the conditions of its deposition on fabrics to form effective and durable xerogel coatings raise the prospects of industrial implementation of the sol-gel method for the coat finishing of cotton fabrics to improve their wear resistance and performance durability.

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