

Surface Coating of Polyester Fabrics by Sol Gel Synthesized ZnO Particles

Merve Küçük, M. Lütfi Öveçoğlu

Abstract—Zinc oxide particles were synthesized using the sol-gel method and dip coated on polyester fabric. X-ray diffraction (XRD) analysis revealed a single crystal phase of ZnO particles. Chemical characteristics of the polyester fabric surface were investigated using attenuated total reflection-Fourier transform infrared (ATR-FTIR) measurements. Morphology of ZnO coated fabric was analyzed using field emission scanning electron microscopy (FESEM). After particle analysis, the aqueous ZnO solution resulted in a narrow size distribution at submicron levels. The deposit of ZnO on polyester fabrics yielded a homogeneous spread of spherical particles. Energy dispersive X-ray spectroscopy (EDX) results also affirmed the presence of ZnO particles on the polyester fabrics.

Keywords—Dip coating, polyester fabrics, sol-gel, zinc oxide.

I. INTRODUCTION

POLYESTER fabric is one of the important fabrics used extensively in clothing, upholstery, and technical textiles. Improving the multifunctional properties by surface coating of this widely used polyester fabric has become a promising study area recently [1]. Providing a variety of properties in a variety of areas, zinc oxide (ZnO) has drawn the attention of the textiles sector. ZnO is one of the major metal oxides. As a wide band (3.37 eV) semiconductor, its low cost synthesis and non-toxic properties, has broadened the use of this metal oxide material. ZnO is a material used in electronic and optoelectronic devices such as solar cells, field emission displays and sensor applications [2]. Also it used for photocatalytic degradation of organic impurities under UV or visible light irradiation [3]. The use of this material in a variety of areas, due to its wide spectrum of properties, has drawn the attention of the textile industry. Improving UV protection, antibacterial and mothproofing activity and biosensor applications has been the focus of recent attempts at attaining multifunctional textiles [4]-[6]. At this point synthesis of ZnO and coating on textile surfaces has taken on a new significance. Sonochemical and microwave assisted synthesis, atomic layer deposition and electroless deposition, are some of the methods used in ZnO synthesis and surface coating [7]-[10].

Another method for fabric surface coating is a sol-gel dip coating method. It is one of the current methods for

M. Küçük is with Istanbul Technical University, Department of Metallurgical and Materials Engineering, Particulate Materials Laboratories (PML), Istanbul, Turkey; (corresponding author, phone: +90 (212) 2853090; fax: +90 (212) 2857347; e-mail: mkucuk@itu.edu.tr).

M. L. Öveçoğlu is with Istanbul Technical University, Department of Metallurgical and Materials Engineering, Particulate Materials Laboratories (PML), Istanbul, Turkey; (e-mail: ovecoглу@gmail.com).

This study was financially supported by 'Istanbul Technical University Scientific Research Project' #38820.

modification of fiber surfaces. This method provides a surface coating with physical properties for desirable multifunctional textile products. Sol-gel synthesis of ZnO particles using zinc salt, results in a rapid reaction at low temperatures, and a narrow size distribution of particles with large surface area properties. Due to the temperature sensitivity of textile fabrics, a low temperature reaction process is very desirable, thus making dip coating a suitable method for surface coating of polyester fibers. In this work, ZnO particles were synthesized using the sol-gel method that involves the dip coating of polyester fabric surfaces.

II. MATERIALS AND METHODS

A. Materials

In this study, undyed 100% polyester fabric was obtained from a local retailer. Zinc acetate dihydrate, trietilamina, isopropyl alcohol, sodium hydroxide, citric acid, Triton X-100 were used for chemical reactions. All the chemicals and reagents were of analytical grades. The polyester fabrics were used after being treated with a washing solution of 2 g NaOH dissolved in 200 ml distilled water, with 0.75 g Triton X-100 and 0.3 g citric acid added in the resulting solution. The washing of the polyester fabric samples was performed with a liquor to goods ratio of 50:1. The fabric samples were immersed into the solution and mixed with a magnetic mixer at 90 °C for 1 h. After rinsing with distilled water, the fabric samples were dried in room conditions.

B. Synthesis of ZnO Particles

ZnO particles were synthesized using the sol-gel method. Zinc acetate dihydrate (0.1 M) was added to the isopropyl alcohol and mixed with a magnetic mixer and heated gradually to 83 °C. At this temperature, 1.4 ml of trietilamin was instilled in the solution. The resulting solution was mixed for 15 minutes in the same conditions and left to cool in room conditions. After adding trietilamin, the solution attained a transparent form, and ZnO particle formation was observed gradually in time.

C. Coating of Polyester Fabrics with ZnO

Polyester fabric samples of 3cm×3cm in size were dip coated. The fabric was immersed into the solution for 15 minutes and then dried in an oven for 15 minutes at 70 °C. This process was repeated twice, and then the fabric samples cured at 100°C for 1 h.

D. Characterization

The characterization methods provided the analysis of the

synthesized ZnO particles and the surface coated polyester fabrics. Average particle size diameter and the polydispersity index (PDI) of the ZnO particles were evaluated employing the dynamic light scattering principle using a NANO-flex (Microtrac) device. The morphology of the pristine polyester fabric and ZnO coated polyester fabric was assessed by FESEM images obtained with a Nova NanoSEM 450. The chemical compositions of the samples were measured by EDX (coupled to FESEM). The FTIR spectrum was recorded on Bruker TM Alpha-T ATR-FTIR spectrophotometer. The analysis conditions were set as resolution 2 cm^{-1} and a frequency range $400\text{--}4000\text{ cm}^{-1}$. The structure of the ZnO particles were characterized by XRD (Bruker™ D8 Advanced Series) spectra with $\text{CuK}\alpha$ radiation ($\lambda=0.154\text{ nm}$, 35 kV and 40 mA) in the 2θ range of $10\text{--}70^\circ$ with a $5^\circ/\text{min}$ scan rate.

III. RESULTS AND DISCUSSION

A. Particle Size Analysis of ZnO Particles Using Dynamic Light Scattering (DLS) Technique

Fig. 1 shows the particle size distribution of the aqueous ZnO solution obtained using the sol-gel process. Average ZnO particle size distribution was obtained at 260.3 nanometers. The PDI was measured as 0.0959, indicating a homogeneous ZnO solution. The PDI value imparts a monodisperse ZnO solution with a narrow distribution. PDI values below 0.1 can be attributed to the high homogeneity of the particle population [11].

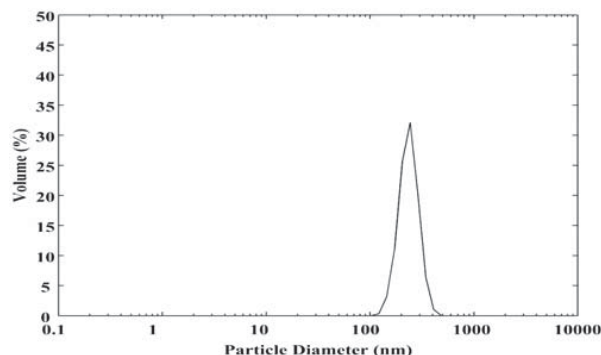


Fig. 1 Particle size distribution of ZnO sol using DLS technique

B. XRD Analysis of ZnO Particles

According to the XRD analysis, zincite ZnO wurtzite structure reflection peaks were observed (JCPDS 36-1451 $a=3.249\text{ \AA}$, $c=5.206\text{ \AA}$). At 2θ value 31.9° (100), 34.5° (002), 36.4° (101), 47.6° (102), 56.6° (110), 63.0° (103), six peaks relevant to hexagonal wurtzite structure were obtained. There have been no diffraction peaks for other impure phases indicating the presence of single phase hexagonal ZnO structures. Single phase ZnO particle synthesis usually requires high temperatures [12]. With the proposed sol-gel process, obtaining single crystal ZnO particles with a low temperature (83°C) synthesis has been made possible.

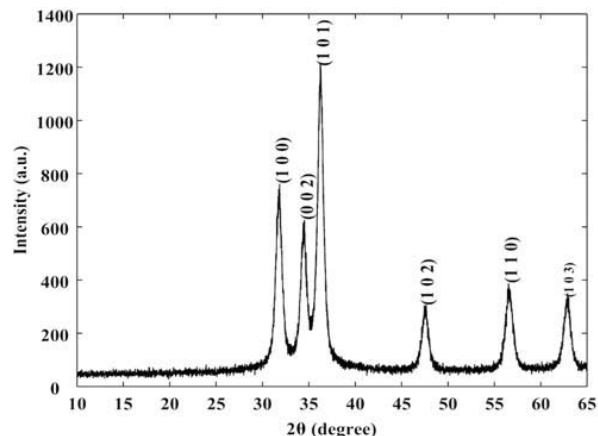
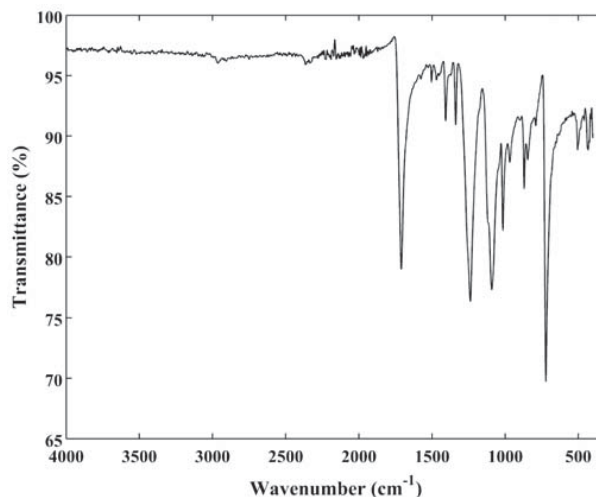


Fig. 2 XRD pattern of synthesized ZnO particles

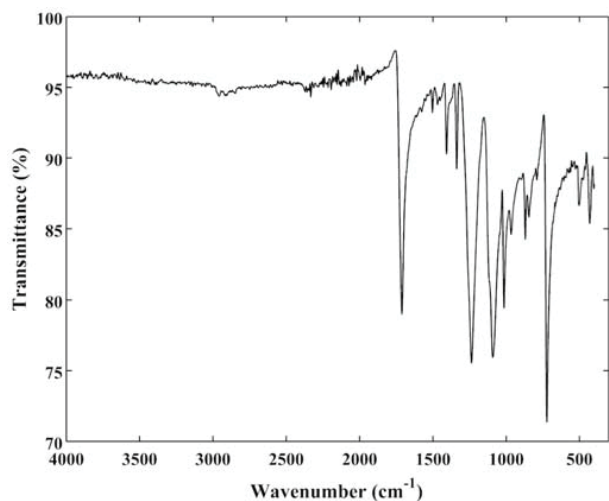
C. ATR-FTIR Analysis of Pristine and ZnO Coated Polyester Fabrics

Fig. 3 shows the ATR-FTIR spectrum for pristine polyester and ZnO coated polyester fabrics. Fig. 3 (a) depicts the characteristic peak values. The sharp peak at 1711.35 cm^{-1} is due to the stretching vibration of the ester group $\text{C}=\text{O}$. The peaks at the polyester benzene ring observed between 1339.04 cm^{-1} - 1016.25 cm^{-1} are due to the stretching peaks of $\text{C}-\text{O}$.

The peaks at 969.19 cm^{-1} at 871.06 cm^{-1} are due to in plane $\text{C}-\text{H}$ ($\text{CH}=\text{CH}$) bending and out of plane $\text{C}-\text{H}$ ($\text{CH}=\text{CH}$) bending, respectively. The weak peak observed at 1503.92 cm^{-1} can be attributed to the stretching vibration of benzene $\text{C}=\text{C}$ [13]. The ATR-FTIR spectrum for both the pristine polyester and ZnO coated polyester fabrics exhibit similar peak positions, except for an intensified peak observed at 432 cm^{-1} (Fig. 3 (b)) corresponding to the stretching vibration of $\text{Zn}-\text{O}$ bending.



(a)



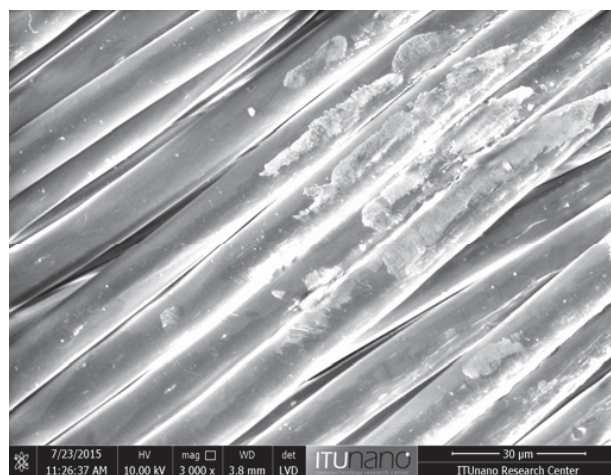
(b)

Fig. 3 ATR-FTIR spectra of (a) pristine polyester fabric (b) ZnO coated polyester fabric

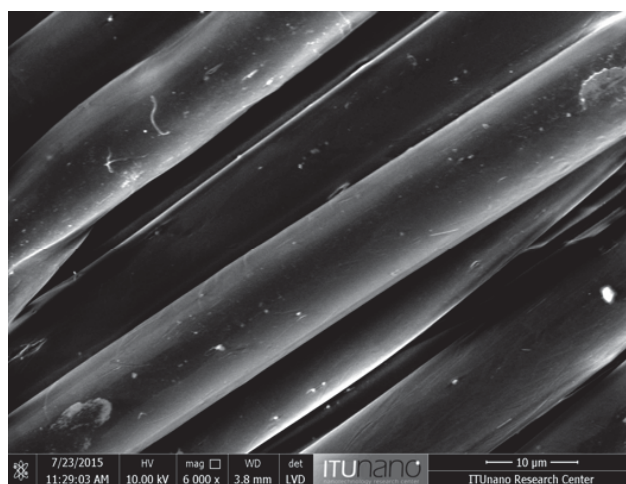
D. Surface Morphology and EDX Analysis Pristine and ZnO Coated Polyester Fabrics

Surface morphology of pristine polyester fabric and ZnO coated polyester fabric were investigated using FE-SEM in Fig. 4. Grooves and fibers on the surface of the pristine polyester fabric are very evident. ZnO particles are uniformly distributed on the coated fabric surface. The particle spread on the surface is in spherical form. Also, particles exhibit an even and dense adhesion to the surface.

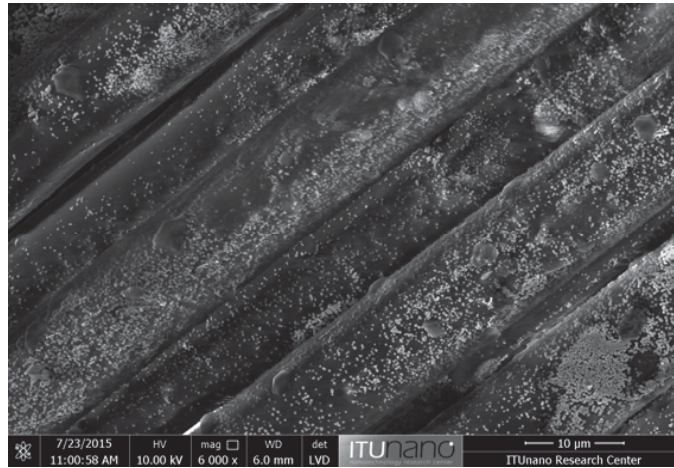
The chemical composition of ZnO coating on polyester fabric was determined by EDX. The results of this analysis for the pristine polyester fabric and ZnO coated polyester fabrics can be seen in Fig. 5 The EDX spectrum of polyester fabric indicates the presence of carbon and oxygen elements. The absence of other elements affirms the surface coating solely of ZnO particles.



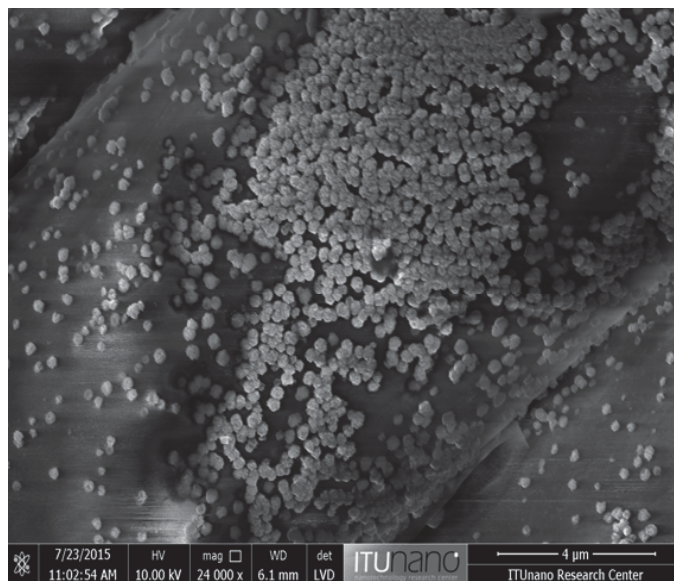
(a)



(b)

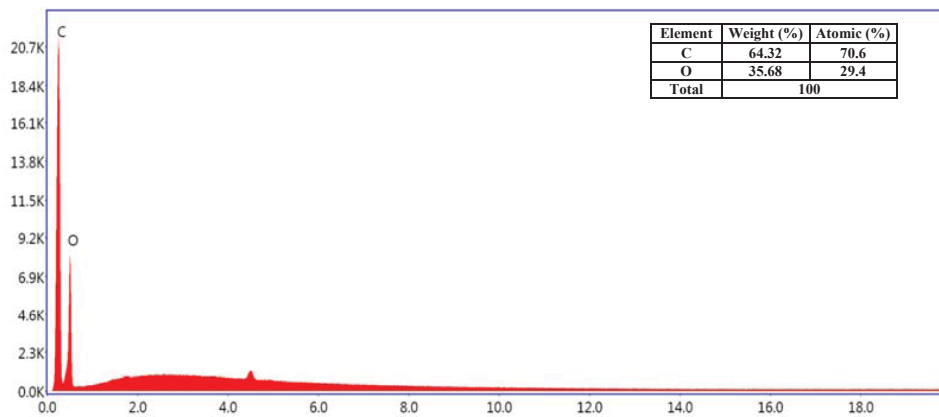


(c)



(d)

Fig. 4 FESEM micrographs pristine polyester fibers (a) low (b) high magnification; ZnO coated polyester fibers (c) low (d) high magnification



(a)

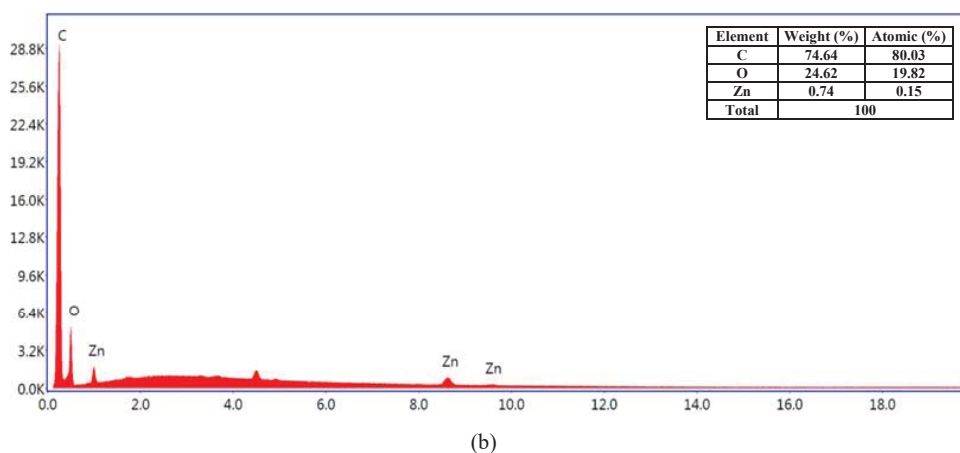


Fig. 5 EDX spectra of pristine (a) pristine polyester fabric (b) ZnO coated polyester fabric

IV. CONCLUSION

ZnO particles were coated on polyester fabric using the sol-gel dip coating method allowing the synthesis of crystal structures for the particles. The average size distribution of the particles was measured as 250 nm. Polyester morphology was determined by FESEM, which revealed a coating with ZnO particles without any damage to the fibers. Also spherical form particles were observed clinging to the surface, with no aggregation.

Generally, sol-gel dip coating is an easy and appropriate method for ZnO synthesis and coating on polyester surfaces, in a possible attempt towards producing functional materials.

REFERENCES

- [1] C. H. Xue, W. Yin, P. Zhang, P.T. Ji, S.T. Jia, "UV-durable superhydrophobic textiles with UV-shielding properties by introduction of ZnO/SiO₂ core/shell nanorods on PET fibers and hydrophobization", *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 427, pp 7-12, 2013.
- [2] A. Hatamie, A. Khan, M. Golabi, A. P. F. Turner, V. Beni, W. C. Mak, A. Sadollahkhani, H. Alnoor, B. Zargar, S. Bano, O. Nur, M. Willander, "Zinc oxide nanostructure-modified textile and Its application to biosensing, photocatalysis, and as antibacterial material", *Langmuir*, 31, 10913-10921, 2015.
- [3] M. Ibanescu, V. Muşat, T. Textor, V. Badilita, B. Mahltig, "Photocatalytic and antimicrobial Ag/ZnO nanocomposites for functionalization of textile fabrics" *Journal of Alloys and Compounds*, 610, pp 244-249, 2014.
- [4] J. Manna, G. Begum, K.P. Kumar, S. Misra, R.K. Rona, "Enabling antibacterial coating via bioinspired mineralization of nanostructured ZnO on fabrics under mild conditions" *Applied Materials Interfaces*, 5, pp 4457-4463, 2013.
- [5] A. Nazari, M. Montazer, M.D. Zahedani, "Mothproofing of wool fabric utilizing ZnO nanoparticles optimized by statistical model" *Journal of Industrial and Engineering Chemistry*, 20 pp 4207-4214, 2014.
- [6] Z.H. Lim, Z.X. Chia, M. Kevin, A.S.W. Wong, G.W. Ho "A facile approach towards ZnO nanorods conductive textile for room temperature multifunctional sensors", *Sensors and Actuators B: Chemical*, 151, pp 121-126, 2010.
- [7] Y.R.C. Urena, S.H.P. Bettini, P.R. Munoz, L. Wittig, K. Rischka, P.N.L. Filho, "In situ sonochemical synthesis of ZnO particles embedded in a thermoplastic matrix for biomedical applications", *Materials Science and Engineering C*, 49, pp 58-65, 2015.
- [8] P. Dou, F. Tan, W. Wang, A. Sarreshteh, X. Qiao, X. Qiu, J. Chen, "One-step microwave-assisted synthesis of Ag/ZnO/graphene nanocomposites with enhanced photocatalytic activity", *Journal of Photochemistry and Photobiology A: Chemistry*, 302, pp 17-22, 2015.
- [9] Y. C. Cheng, K. Y. Yuan, M. J. Chen, "ZnO thin films prepared by atomic layer deposition at various temperatures from 100 to 180 °C with three-pulsed precursors in every growth cycle", *Journal of Alloys and Compounds*, 685, pp 391-394, 2016.
- [10] N. Preda, M. Enculescu, I. Zgura, M. Socol, E. Matei, V. Vasilache, "Superhydrophobic properties of cotton fabrics functionalized with ZnO by electroless deposition", *Materials Chemistry and Physics*, 138, pp 253-261, 2013.
- [11] M. Gaumet, A. Vargas, R. Gurny, F. Delie, "Nanoparticles for drug delivery: The need for precision in reporting particle size parameters", *European Journal of Pharmaceutics and Biopharmaceutics*, 69, pp 1-9, 2008.
- [12] R.N. Khan, S. Riaz, S. Naseem, "Effect of calcination on properties of cobalt doped ZnO nanoparticles", *Materials Today: Proceedings*, 2(10), pp 5765-5770, 2015.
- [13] D. Pasqui, R. Barbucci, "Synthesis, characterization and self cleaning properties of titania nanoparticles grafted on polyester fabrics", *Journal of Photochemistry and Photobiology A: Chemistry*, 274, pp 1-6, 2014.

M. Küçük received her BS from Textile Engineering Çukurova University in 2007. She completed her MS at Kahramanmaraş Sütçü İmam University in 2009 respectively. She worked at Izgi Ltd., 2007-2010, Shirt by Shirt (2010-2011). Since then she is a PhD candidate at Materials Science and Engineering Program, Istanbul Technical University.

M. L. Öveçoğlu graduated from the department of Metallurgy Engineering at the Middle East Technical University in 1979 with an honors degree. He got his Ph. D. Degree from the Materials Science and Engineering Department at Stanford University in 1987 and since 1990 he has been working as a lecturer in the Faculty of Chemistry-Metallurgical, ITU. His main research areas include powder metallurgy, analytic electron microscopy, glass and ceramics and dislocation dynamics.