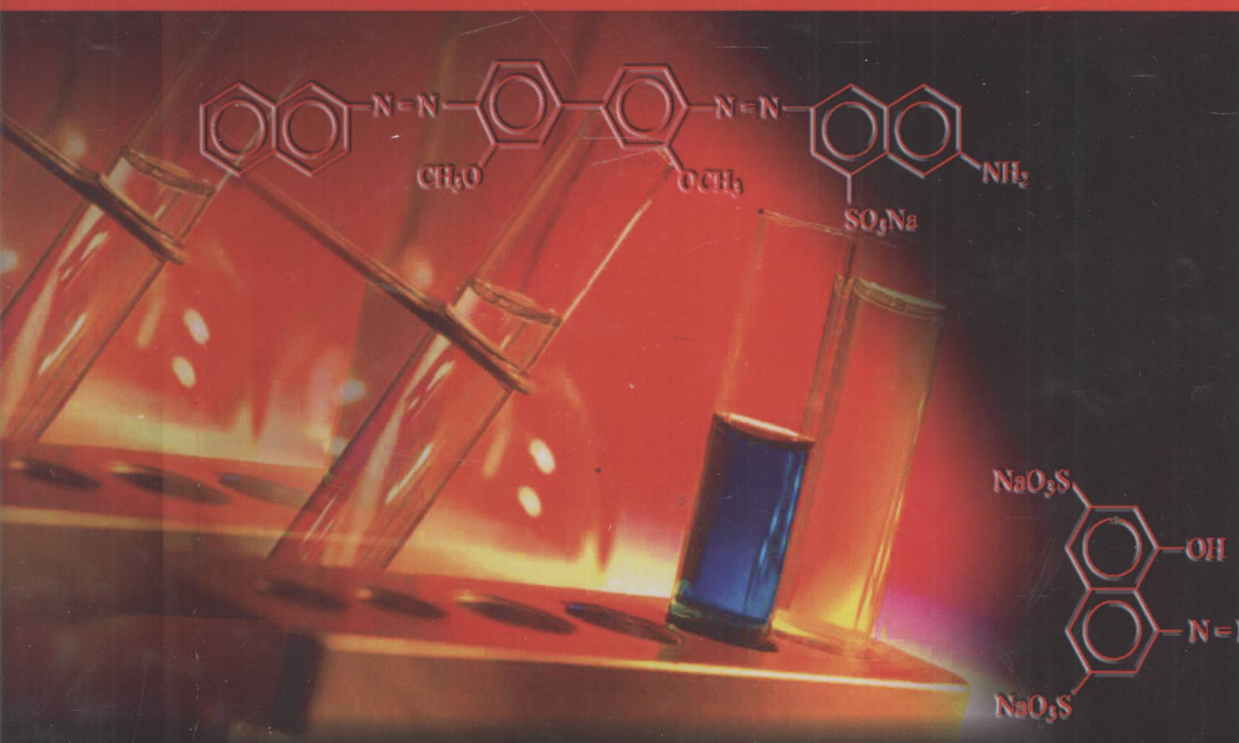
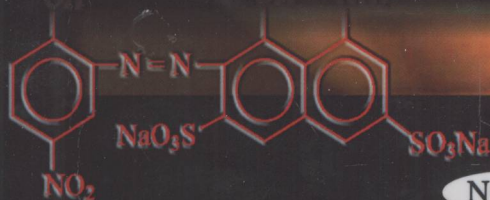


Chemistry of Advanced Compounds and Materials



Nodar Lekishvili • **Gennady E. Zaikov**
Editors



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CHEMISTRY OF ADVANCED COMPOUNDS AND MATERIALS



NODAR LEKISHVILI AND GENNADY E. ZAIKOV
EDITORS



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CHEMISTRY OF ADVANCED COMPOUNDS AND MATERIALS

PREFACE

“Every aspect of the world today even politics and international relations- is affected by chemistry.”

Linus Pauling

The book represents the collection of works of experienced specialists of modern organic and polymer chemistry from Eastern Europe.

The presented papers are dedicated to the following four basic directions:

- I. Development of advanced polymer materials: chemistry, technology and applications:
 - Synthesis of the new type polymers with nontraditional structures and properties;
 - Creation organo-inorganic hybrid materials (nanocomposites) with predefined properties for modern techniques;
 - Development of low-cost resources of raw materials for polymer composites with valuable desired properties;
 - Increase of the quotas of non-metallic materials (especially, polymer composites) in new wares of aerospace, automotive and other industries;
 - Elaboration of the composite materials of high radiation, moisture, aging and chemical resistance from available low-cost resources;
 - Creation of new optical materials based on optically transparent polymers enjoying several advantages over non-polymeric standard materials; Use of the non-traditional physical/-chemical methods for the gradient-formation in optically transparent polymer materials;
 - Elaboration of new electro-conductive polymer composites with wide range of electrical conductivity based on organo-inorganic hybrid materials;
 - Development of non-traditional macromolecular systems composed of α - amino acids - innovative biomaterials
 - Improvement of mechanical properties of polymer composites based on widely used industrial polymers by modification with new type polyfunctional oligomers and polymers;
- II. Nontraditional technologies for manufacturing materials with specific properties;

III. New compounds with predefined properties:

- New organometallic compounds with bioactivity for conservators and antibiocorrosional coatings;
- New biologically active compounds with pharmacophore;
- New biologically active compounds with indole for medical applications
- New bioenergoactivators for use in agriculture.

IV. Chemical Ecology:

- There are discussed many interesting results in the presented areas that will attract attention of readers – specialists of modern materials science and technology.

Editors and contributors of this volume will be glad to receive all the remarks about the book to consider them in the future work.

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PART I.
NONTRADITIONAL MATERIALS AND TECHNOLOGIES

Chapter 1

SUPER-CONDUCTING POLYMER CERAMIC NANOCOMPOSITES: SYNTHESIS AND PROPERTIES

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ABSTRACT

High-temperature super-conducting polymer + ceramic nanocomposites have been synthesized and evaluated. The process of obtaining nano-composites has been investigated. Interphase phenomena at the ceramic-polymer boundary. Some properties of synthesized nano-composites are reported.

High-temperature super-conducting polymer + ceramic nanocomposites were obtained based on $YBaCu_3O_{7-x}$ and a range of binders. Independently of the matrix type used, superconducting (SC) properties of the samples are absent (the Meissner behavior) for the composites formed by hot-pressing at 200 °C. Restoration of superconducting processes occurs only after treatment of samples in a stream of dry oxygen at α -transition temperature of the polymeric binder.

Samples formed at 130 °C possess stable superconducting properties. Polyolefin binders preserve the same critical superconducting transition parameters as the initial ceramic has, while acryl and vinyl homo- and co-polymers enhance the critical SC initiation transition temperature by 1-3 K. Broadening of the SC transition temperature interval of the composites at higher temperatures of formation (≥ 160 °C) is associated with thermo-oxidative degradation of the polymeric binder. Mitigation of the thermal degradation of the matrix (reducing pressing duration, addition of anti-oxidants) allows maintaining the critical SC transition parameters of the initial ceramic.

Dynamic mechanical properties (storage modulus, phase angle) of ultra highmolecular polyethylene (UHMWPE) have been investigated. There is a strong interaction of the binder with the surface of the ceramic grains. Changes of packing and

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structure of the macromolecules occur at the ceramic-polymer interface. This is confirmed by melting enthalpy measurements of SC the composites of different filler content.

Scanning electron microscopy (SEM) studies show that the ceramic grains are evenly covered by the binder for both amorphous and crystalline polymers. EPR (electron paramagnetic resonance) spectra of polymer-ceramic composites show that the intensity of the EPR signals of Cu^{2+} (1) depends on the nature and the content of binder. Mn, Co, Zn and Ni containing super-conducting composites have been obtained by polymerization.

INTRODUCTION

High-temperature super-conducting polymer ceramic composites can be obtained both by conventional hot-pressing of a mixture of ceramic and ready-made high-molecular binder, and by the polymerization filler method [1, 2]. Hot pressing of the $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{7-x}$ oxide ceramic and super-high-molecular polyethylene mixture at 200°C annihilate the SC properties. These properties are restored only after the treatment of samples in dry oxygen stream [2]. For $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{7-x}$ superconductive polymer-ceramic composites with different polymeric binders, an increase of the superconducting transition by 1-3 K was observed previously [1-4]. The increase is due to interaction of polymer chains with surfaces of the ceramic grains. One could expect that such an interaction should change the packing and structure of the polymer chains, as well as the conformation at the interphase. In this work interphase phenomena at the ceramic-polymer boundary are investigated for superhighmolecular polyethylene + $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{7-x}$ ceramic. The influence of crystalline binders on the valence state of Cu^{2+} (1) in the ceramic has been investigated as well.

EXPERIMENTAL

$\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{6.92}$, $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{6.97}$ had the following characteristics: critical transition temperature of initiation of superconductivity, $T_i - 91.6^\circ\text{K}$ and 93°K , while its width $\Delta T_i - 6$ degrees and 6.5 degrees accordingly were used as a high temperature oxide ceramic.

A wide variety of porous polymer binders were used, such as high-pressure polyethylene (HPPE) with $T_{\text{melt}}=15-108^\circ\text{C}$; super-high-molecular polyethylene (SPE) of the U-506-000 brand with $T_{\text{melt}}=128-135^\circ\text{C}$; isotactic polypropylene (PP) with $T_{\text{melt}}=167-171^\circ\text{C}$, isotactic polybutylene (PB) having $T_{\text{melt}}=135^\circ\text{C}$; co-polymer of ethylene with tetrafluoroethylene (CETFE) with $T_{\text{melt}}=265-278^\circ\text{C}$; Polyvinyl idenfluoride (PVF) with $T_{\text{melt}}=171-180^\circ\text{C}$; polyvinyl alcohol (PVA) with $T_{\text{vitrification}}=85^\circ\text{C}$; polyformaldehyde (PFA) with $T_{\text{melt}}=173-180^\circ\text{C}$; polymethylmetacrylate (PMMA) with $T_{\text{vitrification}}=100-105^\circ\text{C}$; polystyrene (PS) with $T_{\text{vitrification}}=98-102^\circ\text{C}$ as well as copolymer of methylmetacrylate with styrene (CPSM) with of styrene in the copolymer 80, 60 or 40 mole %.

Vitrification temperatures of the used co-polymers with methylmetacrylate (MMA) are between $85-97^\circ\text{C}$, a result of non-homogeneity of the composite.

Temperature of melting of HPPE, SPE, PP, as well as vitrification temperature of PMMA, PS, as well as CPSM were determined by DSM-3 scanning calorimeter (*company Pushchino*) at the heating rate 1.0 K/minute.

PMMA, PS and CPSM were synthesized by radical polymerization method in ethyl acetate solution with the use of benzoyl peroxide as an initiator ([Monomer]=3.5 mole/l, [benzoyl peroxide]= 1.5×10^{-2} mole/l, $T=75^{\circ}\text{C}$, duration – 10 h), industrial brands of polymers and co-polymers of styrene with methylmetacrylate were used as an alternative kind of binders.

"Irganox"-1010 and NG-2246 (*from Chemical company, Okhtinsk*) in 0.5 and 5 mass per cent from polymeric binder were used as antioxidants.

Articles made from polymer-ceramic superconducting composites were produced in the following manner: preliminarily mixed powders of high-temperature superconducting ceramic and polymer in definite proportions were poured into a pre-heated mould to form articles at 100 MPa pressure during 4 or 30 min (conventional forming times). Then the mould was cooled down to circa 40°C during 15 min to obtain ready-made articles.

Structural parameters of high-temperature superconducting ceramic and composite were determined by X-ray analysis using: DRON 2.0 ($\lambda\text{CuK}\alpha$) (*company LOMO*).

Critical transition temperature into superconducting state of the samples, were determined by dynamic magnetic susceptibility method. A laboratory installation used had the following parameters: sinusoidal magnetic field with 10 mE amplitude, frequency 1 kHz, temperature determination range: 80 – 100 K, precision: ± 0.1 K.

Thermo-oxidation destruction of polymeric binders was investigated by derivato-graphic method on the instrument "MOM" Q-1500 at the heating rate 3.2 K/minute.

Catalytic polymerization of ethylene on surfaces of the grains of powdery oxide ceramic was performed at room temperature in hexane with vigorously agitation at 100 rpm.

Alkyl aluminum was used as a polymerization co-catalyst. The polymer-ceramic composites so obtained were rinsed with absolute alcohol and dried at 700°C up to constant weight to remove the remains of alkyl aluminum. Specimens were pressed at 150°C in required dimensions for the measurement of superconducting properties.

As filler, oxide ceramics $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{6.97}$ with the following characteristics have been used: critical temperature of transition into SC state: 93 K, width: 60, dispersity (0.1-50) μm . Polymer-ceramic composites were prepared as described previously [1-4].

Superconductive composite samples based on SHPE have the form of plates of dimension: $3 \times 1 \times 0.1 \text{ cm}^3$ (matrix: filler=100:0; 85:15; 50:50; 15:85 mass ratios). These samples are used for studying the dynamic mechanical properties.

Mechanical relaxation properties of the SHPE composites have been measured using a Du Pont dynamic mechanical analysis (DMA) instrument under amplitudes of oscillations 0.1 - 0.2 mm. Calorimetric measurements have been conducted on a DSM-3A differential scanning calorimeter.

Structural peculiarities of superconductive polymer-ceramic composites have been investigated using a scanning electron-microscope "Tesla-301" (*Czechoslovakia*)

Frontal polymerization of the powdery mixture of Co-, Ni-, Mn-containing metal-complexes of the monomers with the $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{6.97}$ was carried out in vertically positioned glass reactors. Heat (200°C) impulse at the bottom side of the reactor has initiated front wave

propagation from down to up. Metal complex monomers were synthesized from the Co, Ni, Mn nitrates with acryl amide according to [5].

SUPERCONDUCTING PROPERTIES OF POLYMER-CERAMIC COMPOSITES OBTAINED BY THE HOT PRESSING METHOD

We have shown before [2] that formation of polymer-ceramic composites based on super-high-molecular polyethylene at 200 °C, $\tau_f = 30$ min yields samples possessing no superconducting properties (the Meissner effect is absent). Nevertheless, superconducting properties are restored after treatment in dry oxygen stream at the temperature of α -transition of the polymeric binder. The same phenomenon is observed at similar conditions of formation of the composites, independent upon the chemical composition of the polymeric matrix, see Table 1. Thus, the absence of superconducting properties depends on release of free oxygen on the surface of the oxide ceramic grains, as well as on irreversible reactions of thermo-oxidative degradation of the polymeric matrix. Similar results have been obtained in the presence of Irganox a polymeric antioxidant supporting our conclusion Rudin and coworkers have shown [6], that antioxidant additives in the polymeric matrix substantially reduce the rate of oxidation destruction in the polymers. Apparently introduction of minute quantities (0.5 weight % of the binder) of antioxidant decreases the rate of the oxidation reactions and consequently retards the depletion of the oxygen on the surfaces of the ceramic grains. Actually, in the compositions with small amounts of the antioxidant, right after pressing, without the stage of restoration, the samples exhibit superconducting properties, see Table 1.

As expected Table 1 tells us that restoration of the superconducting properties by dry oxygen the beginning transition temperature as well as its end point depends on the nature and chemical composition of the polymeric binder.

Table 1. Superconducting properties of composites based on $\text{YBa}_2\text{Cu}_3\text{O}_{6.97}$ ceramic ($T_f = 200$ °C, $\tau_f = 30$ min)

Composition	Ceramic/binder weight ratio	T_1 / K	T_c / K	Comments
SHMP + (123)	80 : 20	96	84	Obtained after the stage of restoration
	85 : 15	96	84	
	85 : 15	96	84	
RPE + (123)	80 : 20	94	80	— ” —
F-40 + (123)	75 : 25	96	77	— ” —
PB + (123)	80 : 20	96	83	— ” —
PVF + (123)	85 : 15	90	75	— ” —
PFA + (123)	80 : 20	88	76	— ” —
SHMP + Irganox + (123)	80 : 20	96	89	Obtained without restoration
RPE + Irganox + (123)	80 : 20	94	85	— ” —
PVA + Irganox + (123)	85 : 15	90	80	— ” —

The superconducting critical temperature increases by 2 - 3 K ° except for PVA, PMMA and PFA, physical interactions of separate fragments of the macromolecules of the binder with the surface of the (123) ceramic, up to intercalation of these fragments into interstitial layers of the ceramic grain [7] or anchoring there free oxygen positions. For composites with poly(vinyl alcohol) the observed decrease of T_c by 5 K ° suggests possible interactions of some fragments of the polymeric chains with the surfaces of the ceramic. In this case OH groups, like in water [8], alcohol or in weak acids [9], while reacting distort the orthorhombic phase in nearby-surface layers of the ceramic grains.

For the polyformaldehyde containing composites we see considerable T_c decrease, it can be explained by a propensity of polyformaldehyde towards thermo degradation, which enhances the consumption of oxygen in the ceramic. Moreover, even at comparatively low temperatures [10] there is a reaction of de-polymerization with release of gaseous formaldehyde. This in turn can adversely affect the superconducting properties.

THERMO-OXIDATION DESTRUCTION OF THE POLYMERIC BINDER AND COMPOSITES BASED ON IT

Results of the derivatographic analysis of the samples under investigation are presented in Table 2 and Figure 1, Figure 2.

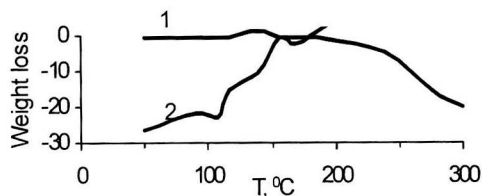


Figure 1. Weight loss (per cent of the super-high-molecular-polyethylene) (curve 1) and differential thermal assay (curve 2) via thermo oxidation destruction of the super-high-molecular-polyethylene. Heating rate is 3.2 degrees per min.

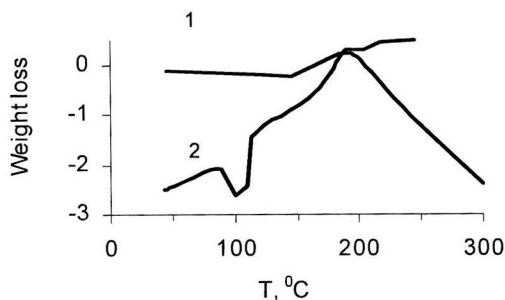


Figure. 2. Weight loss (per cent of the binder) (curve 1) and differential thermal assay (curve 2) of the super-high-molecular-polyethylene (SHMP) + $Y_1Ba_2Cu_3O_{6.97}$. The composition of the composite: SHMP: $Y_1Ba_2Cu_3O_{6.97}$ = 20:80 (in mass ratio of the monomer). Heating rate is 3.2 degrees per min.