



QATTIQ ION O'TKAZGICH: POLIANILINLANGAN NANOTRUBKALAR TIO₂ YOKI ZRO₂ MATRITSASIDAGI (PANI).

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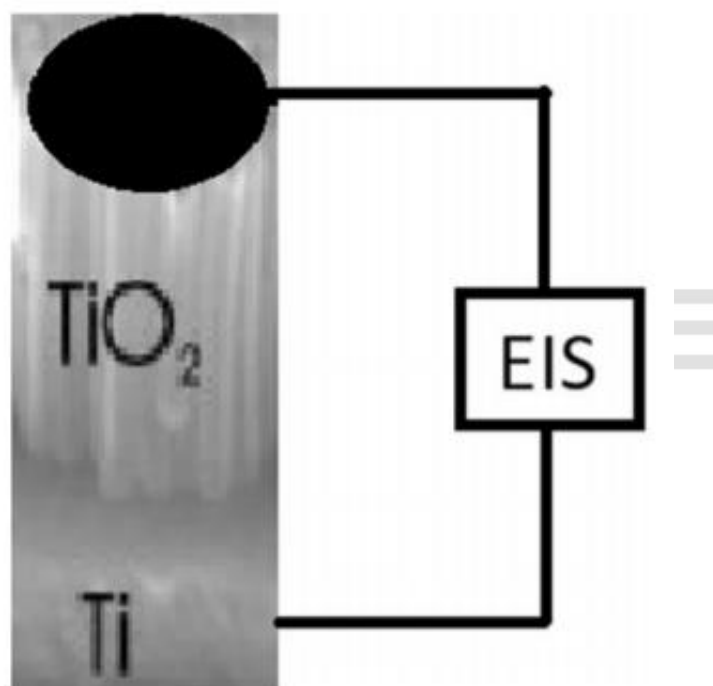
Nanokompozit materiallar.
Polimer elektrolitlar. Proton
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ABSTRACT

Nanokompozitli qattiq ion o'tkazgich Zr yoki Ti metallini elektrokimyoviy anodlash yo'li bilan ishlab chiqarilgan sirkoniya yoki titaniya nanotrubka massivlarini proton o'tkazuvchi Polianilin (PANI) ionomeri bilan infiltratsiya qilish orqali tayyorlangan. Olingan material skanerlash elektron mikroskopiyasi, rentgen nurlari diffraksiyasi va infraqizil spektroskopiya yordamida nanotrubkalar matritsaning ionomer bilan muvaffaqiyatli to'ldirilganligini ko'rsatib tavsiflangan. Empedans spektroskopiyasi infiltratsiyadan so'ng o'tkazuvchanlikning bir necha darajaga ko'payishini aniqladi; bundan tashqari, TiO₂ ning empedansi PANI nanokompoziti nisbiy namlikka juda sezgir. Ushbu ion o'tkazuvchan nanokompozitlarning mumkin bo'lgan qo'llanilishi qattiq holatdagi namlik sensorlari yoki heterojen katalitik materiallarni o'z ichiga oladi.

Past o'lchamli qattiq ion o'tkazgichlarning ion o'tkazuvchanligi [1-3] maxsus tuzilma turlarida, ayniqsa bir o'lchovli o'tkazuvchanlik kanallarini o'z ichiga oladi (masalan, Hollanditlar [2]) yoki ikki o'lchovli panjara tekisliklari (masalan, balumina [4]), ko'pincha juda anizotropdir. Kompozit materiallar ham tayyorlanishi mumkin [5]; Bunday holda, kompozit struktura anizotrop o'tkazuvchanlik xususiyatlarini ko'rsatish uchun ion o'tkazuvchanlik fazasining yuqori tartibli tartibiga ega bo'lishi kerak, masalan, mezoskopik ion o'tkazuvchan heterostrukturalarda.[6]. Yuqori yo'naltirilgan o'tkazuvchanlik yo'llari bo'lgan nanokompozit ionli o'tkazgichlar, asosan, ion o'tkazuvchanlik fazasi bilan to'ldirilgan nanotrubkalar shablonlardan foydalangan holda tuzilishi mumkin.[7]. Yuqori anizotropik ion o'tkazuvchanlik xususiyatlarini o'rganishning asosiy jihati bilan bir qatorda, past o'lchamli qattiq ion o'tkazgichlarining potentsial qo'llanilishi keng tarqalgan, shu jumladan qattiq holatdagi sensorlar [8] yoki heterojen kataliz [9]. Ushbu ishda biz o'z-o'zidan tashkil etilgan ZrO₂ dan foydalanamiz yoki TiO₂ nanotrubkalar proton o'tkazuvchi polianilin (PANI) polimerni cho'ktirish uchun shablon sifatida [10-13]. Pristine ZrO₂ yoki TiO₂ nanotrubkalarni

sirkoniy yoki titan metall plyonkalarini elektrolitlar tarkibida ftorid-ionda elektrokimyoviy anodlash yo'li bilan tayyorlash mumkin.[14,15]. Cheklangan kuchlanishli ftorid ionlarini o'z ichiga olgan organik va suvli elektrolitlar tizimlarida o'z-o'zidan buyurtma qilingan sirkoniya nanotrubkalarining elektrokimyoviy o'sishi ma'lumotnomalarda xabar qilingan.[6-18]. Guo va boshqalar. [19] elektrolitlarni o'z ichiga olgan xloridida Zr ni anodlash yo'li bilan o'z-o'zidan tashkil etilgan sirkoniya g'ovakli qatlamning shakllanishi va o'sishini o'rganib chiqdi. G'ovakli qatlamlarning morfologiyasiga elektrokimyoviy sharoitlar, shu jumladan Cl⁻ konsentratsiyasi ta'sir ko'rsatdi. Tegishli sharoitlarda anodizatsiya yo'li bilan silliq va to'g'ri devorlarga ega, diametri 250 dan 300 nm gacha va uzunligi 33 mkm bo'lgan sirkoniya nanotrubkalarini ishlab chiqarish mumkin. Sakai va boshqalar. [20] sulfat kislota o'zgartirilgan nanotitaniyaning proton o'tkazuvchanlik xususiyatlarini xabar qildi. Proton o'tkazuvchanligi, adsorbsiyalangan sulfat guruhlari miqdori deyarli bir xil bo'lganida, suvli sulfatlangan nanotitaniki bilan deyarli bir xil edi. Yuqori tartibli va juda nozik TiO₂ nanotrubkalar o'zgartirilgan tezkor anodizatsiya usuli bilan ham ishlab chiqarilgan [21]. Polianilin-titaniya nanotrubka gibridlari polianilinni yaxshi tekislangan titaniya nanotrubkalariga elektrodepozitsiya qilish, shuningdek elektrokimyoviy pasaytirish va cho'ktirish usuli bilan tayyorlangan.[22].

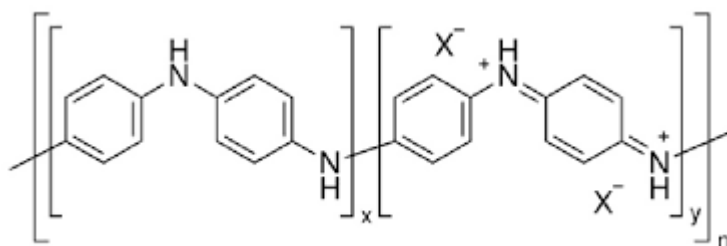


1-rasm. Kontaktning joylashtirish sxemasi.

Nanotrubkalar ZrO₂ xona haroratida yuqori qarshilikka ega [23], TiO₂ esa nanotrubkalar n-tipli yarim o'tkazuvchanlikni ko'rsatadi [24]. Sirkoniya nanotrubkalarini elektrokimyoviy kamaytirish TiO₂ nanotrubkalariga qaraganda ancha qiyin, chunki Zr⁴⁺ ionlari Ti⁴⁺ ionlariga qaraganda kamroq osonlik bilan kamayadi [24]. Shu ma'noda, sirkoniya nanotrubkalari izolyatsiyalovchi matritsaning modelini va titaniya nanotrubkalari xona haroratidagi ion o'tkazgich bilan infiltratsiya qilish uchun model yarim o'tkazgich matritsasini ifodalaydi. Infiltratsiya uchun namunaviy qattiq ion o'tkazgich ishlatilgan: Polianilin (PANI) xona haroratida proton o'tkazuvchi ionomer bo'lib, ko'pgina xususiyatlar, shu jumladan proton o'tkazuvchanligi juda yaxshi ma'lum.[25-27]. Nanofaza bilan ajratilgan polimer

strukturasiining kanallarida suv molekularining mavjudligi ionlarni tashish uchun suv vositachiligini ta'minlaydi.[26].

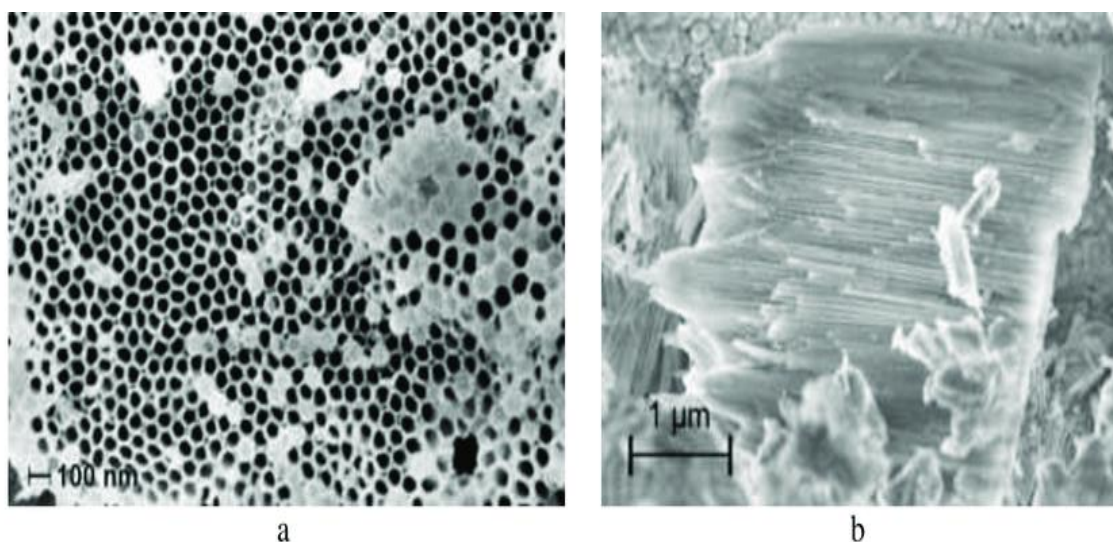
Bunday yuqori anizotropik nanokompozitlarni amalga oshirish uchun birinchi hal qiluvchi qadam nanotrubkalar shablonlarni to'liq to'ldirish imkonini beruvchi yuqori namlovchi ionomer eritmasini tayyorlashdir. Muhim jihat ZrO_2 ni yoki TiO_2 yuzalarini mukammal namlash qobiliyatiga ega ionomer uchun mos erituvchini topishdir. [28]. Olingan nanokompozit struktura ionli o'tkazgichning yuqori nisbati va yuqori yo'naltirilgan o'tkazuvchanlik yo'llarini taqdim etadi. Namlikka sezgir detektorlarni amalga oshirish uchun biz yarim o'tkazgich titaniya matritsasi bilan ushbu nanokompozitlarning ion o'tkazuvchanligini suv bug'ining bosimiga bog'liqligini o'rganamiz. Protonning harakatchanligi proton-katalizlangan reaksiyalar uchun model geterogen katalizatorlarni amalga oshirish imkoniyatini ham ochadi.



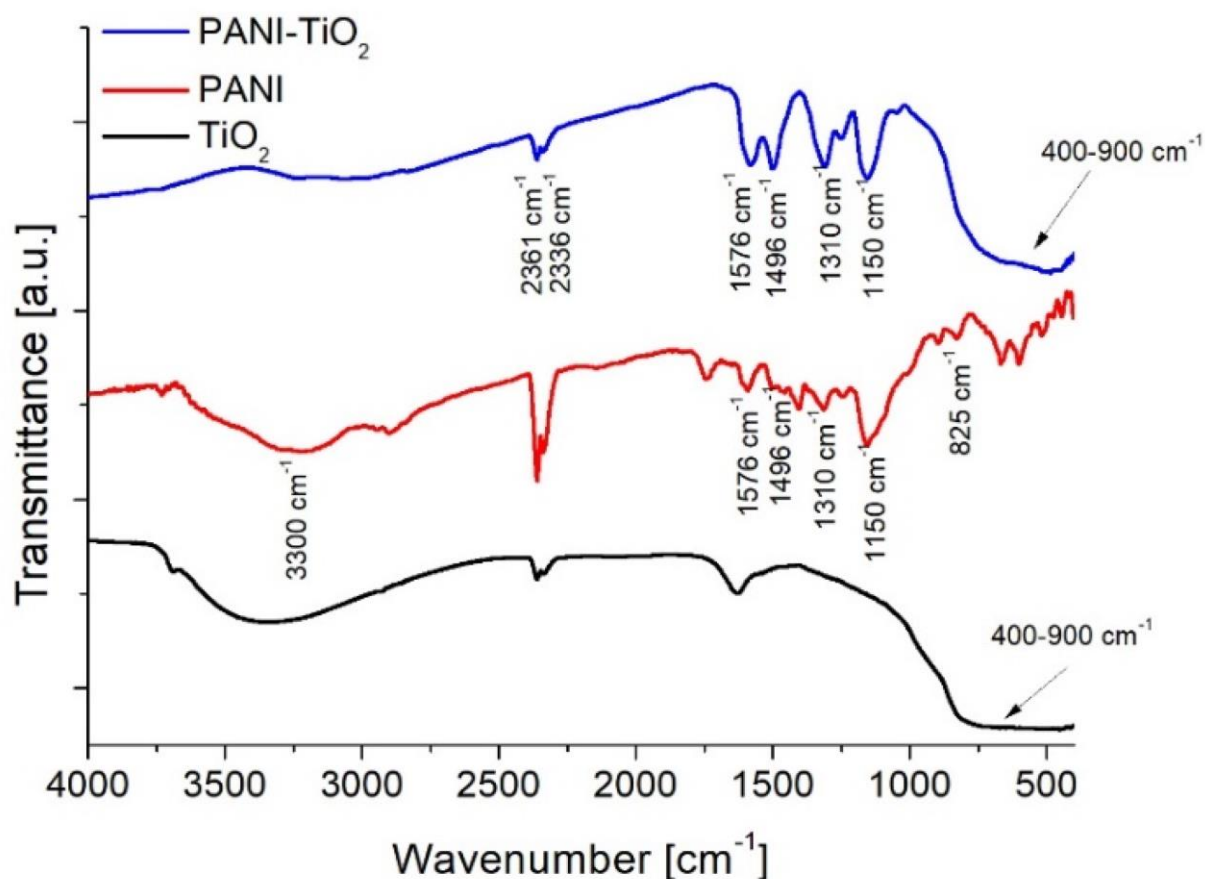
2-rasm. Polianilin (PANI) ning strukturasi

Materiallar va usullar:

Taxminan 2 mkm qalinlikdagi Zr va Ti metall plyonkalari ilgari xabar qilinganidek, Si substratlarida katodik purkash orqali to'plangan.[14]. Zr plyonkalari xona haroratida 0,35 M NH_4F da an'anaviy ikki elektrodli konfiguratsiya yordamida 10 daqiqa davomida 40 V kuchlanishda 5 vol% suv o'z ichiga olgan glitserindagi eritmasida anodik oksidlangan. Ti plyonkalari og'irligi 1,3% wt va 2 g% suv 60 V da 3 soat davomida NH_4F ni o'z ichiga olgan glitserin eritmasida anodlangan. Elektrodlar yuzasidan kislorod pufakchalarining ajralishini rag'batlantirish uchun glitserin eritmasi biroz aralashtirildi (100 rpm), natijada nanotrubkalarining morfologiyasi va umumiy sifati yaxshilandi. Qarshi elektrod sifatida platina panjara ishlatilgan [14]. ZrO_2 yoki TiO_2 nanotrubkalar impedans o'lchovlari uchun orqa elektrod bazasida anodizatsiyadan keyin qolgan sirkonyum yoki titanium metall sifatida ishlatiladi. Anodizatsiyadan so'ng, nanotubkalar deionizatsiyalangan suv bilan mo'l-ko'l yuvilgan va vakuum ostida 80 ° C da 2 soat davomida quritilgan. Oldin xabar qilinganidek, polianilin (PANI) konsentrlangan sulfat kislota bilan 50°C da reaksiyasi natijasida tayyorlangan. [29,30]. Infiltratsiya 7,1 mg (PANI) ni 10 ml dimetilformamidga (DMF) eritish orqali amalga oshirildi.



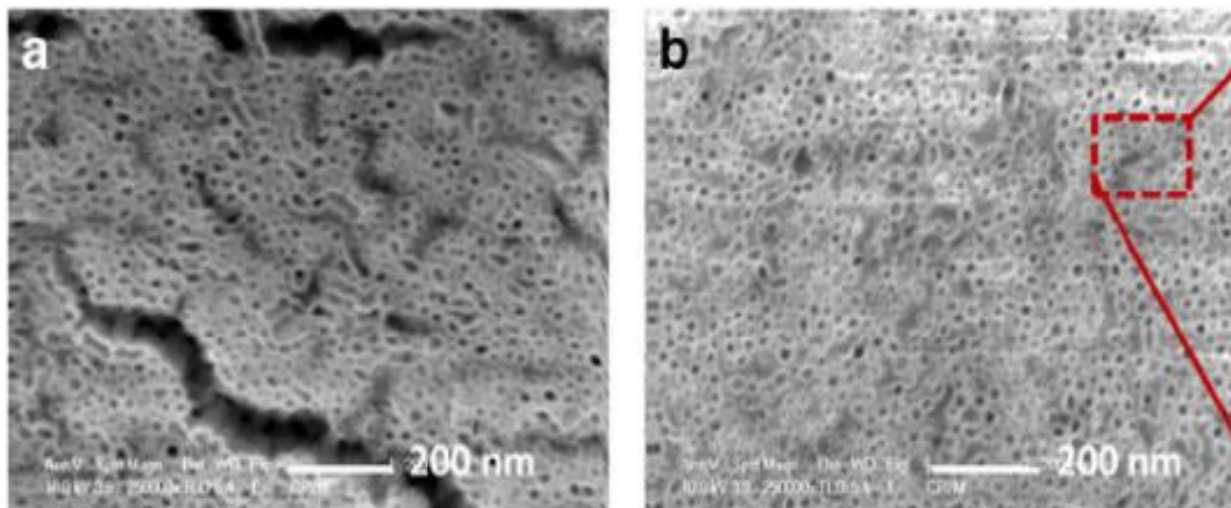
3-rasm. Anodlangan ZrO_2 qatlamining sirt (a) va lateral ko'rinishi (b) ning SEM tasvirlari. Bir tomchi bu eritma $\sim 0,5 \text{ sm}^2$ ga cho'ktiriladi sirkoniya yoki titaniya nanotrubkalarining 1 tomchi ($\sim 0,1 \text{ ml}$) eritmaga kiritilgan PANI miqdori nanotrubkalarni to'liq to'ldirish uchun yetarli bo'lishi uchun hisoblab chiqilgan. Na'munalar Siemens D5000 difraktometri yordamida $CuK\alpha$ nurlanishi bilan rentgen nurlari difraksiyasi (XRD) orqali tahlil qilindi. Nanokompozitning sirt morfologiyasi va tarkibi skanerlash elektron mikroskopi (SEM, Hitachi, S-570) orqali o'rganildi. Furiye-transformatsion infraqizil (FTIR) spektrlari 4000 dan 400sm^{-1} gacha uzatish rejimida qayd etilgan. Bruker Equinox 55 spektrometri yordamida to'lqin raqami. Empedans diagrammalari Parstat 2273 yoki 4000 impedans analizatorlari yordamida 1 Hz dan 100 kHz gacha chastota diapazonida 20 mV signal amplitudasi bilan qayd etilgan. Orqa aloqa qolgan Ti metall plyonkasida olingan; oldingi kontakt 6 mm diametrli zanglamaydigan po'latdan yasalgan disk edi. Ma'lumotlarning takrorlanishi va to'g'riligini tekshirish, kontaktning maydoni va holatini o'zgartirish uchun bir nechta tajribalar o'tkazildi. TiO_2 bilan impedans o'lchovlari nt-SPEEK nanokompozitlari turli nisbiy namlik (RH) ostida ham amalga oshirildi. Quruq sharoitlar uchun na'muna bir kechada 100°C da pechda quritilgan va quruq qutiga yig'ilgan. Natijalar va muhokama:



4-rasm. TiO_2 , polianilin (PANI) va TiO_2 /PANI ning (FTIR) spektrlari.

Nanokompozitni tayyorlashning birinchi muhim bosqichi nanotrubkalar matritsa uchun mukammal namlash xususiyatiga ega erituvchini topish edi. Sulfat kislotasi H_2SO_4 va metan sulfonik kislotasi turli xil keramik-uglerodli nanotrubkalar kompozitlarini namlashi haqida xabar berilgan. ZrO_2 [23,24]. Ma'lumki, sulfonlanish polimerning gidrofilligini oshiradi va kationlarni tashishga yordam beradi. Polimerning gidrofilligi va eruvchanligi sulfonlanish darajasi bilan ortadi. 60% dan past sulfonlanish darajasida PANI faqat sulfat kislotasi kabi kuchli kislotalarda eriydi. Sulfatsiyalanish darajasi 80% dan yuqori bo'lsa, PANI polimeri metanol, DMFA va issiq suvda eriydi. 20 dan 40% gacha bo'lgan suvni qabul qilish mexanik barqaror polimerni ushlab turganda yuqori darajadagi ion o'tkazuvchanligi uchun eng mos keladi. Empedans spektroskopiyasi bilan o'lchangan gidratlangan PANI membranalarining proton o'tkazuvchanligi oshganini ko'rish mumkin. Eritma konsentratsiyasi bir tomchi eritmada nanotrubkalar hajmini yuqorida ionomer qatlami hosil qilmasdan to'liq to'ldirish uchun yetarli PANI miqdoriga ega bo'lishi uchun moslashtirildi. PANI bilan to'ldirilishi kerak bo'lgan ZrO_2 va TiO_2 nanotrubkalari hajmning avval xabar qilingan o'lchamlari bo'yicha hisoblanishi mumkin. Ushbu ishda tasdiqlangan nanotrubkalar o'rtacha ZrO_2 nanotrubkaning uzunligi taxminan 1 mkm; tasvirda ko'rinadigan nanotrubkaning diametri 40 nm atrofida. Taxminan 5 nm devor qalinligi ilgari xabar qilingan edi [14]. TiO_2 uchun, o'rtacha uzunligi va diametri mos ravishda 1,5 mkm va 100 nm, devor qalinligi esa taxminan 10 nm. Ionomer bilan to'ldirilgan ZrO_2 nanotrubka massivlarini 3-rasmda ko'rish mumkin. Haqiqatan ham tepada ionomer qatlami hosil bo'lmaydi va ko'pchilik nanotrubkalar to'ldirilgan ko'rinadi, buni turli kontrastlardan xulosa qilish mumkin. Ionomer bilan to'ldirilgan nanotrubkalarining XRD namunasi ZrO_2 ning qisman kristalli tuzilishini tasdiqlaydi. Avval xabar qilinganidek,

monoklinik va tetragonal fazalarni o'z ichiga olgan nanotrubkalar [23, 32]; polimer bilan to'ldirilgan. Nanotrubkalarda Bragg burchagi $2\theta = 20^\circ$ atrofida kuzatilgani keng aks etgan, avval muhokama qilinganidek, to'liq amorf PANI tufaylidir [33]. Toza va ionomer to'ldirilgan ZrO_2 nanotrubkalarining FTIR spektrlarini ko'rsatadi. 900 dan 1500 cm^{-1} gacha bo'lgan cho'qqilar gidroksilga tegishli sirt guruhlari. 1630 cm^{-1} da cho'qqisi avval NH_4^+ ionlariga tayinlangan edi, chunki sirkoniya nanotrubka massivlari NH_4F ni o'z ichiga olgan elektrolitda tayyorlangan [9]. Ionomer bilan bog'liq cho'qqilar (1-jadval) oldingi hisobotlarga mos keladi [30].



5-rasm. To'ldirilmagan va (PANI) bilan to'ldirilgan ZrO_2 nanotrubkalarining SEM tasvirlari

Xulosa

Nanokompozit qattiq ionning tayyorlanishi va xossalari o'tkazgich, PANI ning TiO_2 yoki ZrO_2 ga infiltratsiyasi orqali amalga oshiriladi nanotrubkalar haqida xabar berilgan. Mikro tuzilma va faza tarkibi SEM, XRD va FTIR spektroskopiyasi bilan tasdiqlangan. Ion o'tkazuvchi polimerning infiltratsiyasi natijasida na'muna qarshiligining kuchli pasayishi impedans spektroskopiyasida aniqlanadi.

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