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ION-PLAZMA VA LAZER TEXNOLOGIYALARI INSTITUTI

ISMATOV ASKAR ANVAR O‘G‘LI

KATALIZATOR-BUFER QATLAM INTERFEYSIDA NANOZARRACHALAR SHAKLLANISH JARAYONLARI VA ULARNING UGLEROD NANONAYCHALARI O‘SISHIDAGI TA’SIRI

01.04.12 – Nanomateriallar fizikasi va texnologiyasi

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Ilmiy rahbar:	**Ashurov Xotam Baxronovich** texnika fanlari doktori, professor
Rasmiy opponentlar:	**Tajibayev Ilhom Ibrohimovich** fizika-matematika fanlari doktori, yetakchi ilmiy xodim, **Haqberdiyev Elshod Olmosovich** fizika-matematika fanlari bo`yicha falsafa doktori, katta ilmiy xodim,
Yetakchi tashkilot:	Mirzo Ulug‘bek nomidagi O‘zbekiston Milliy universiteti

Dissertatsiya himoyasi Ion-plazma va lazer texnologiyalari instituti huzuridagi ilmiy darajalar beruvchi DSc.05/2025.27.12.FM/T.09.01 raqamli Ilmiy kengashning **2026 yil «3» dekabr soat 16^{00}** dagi majlisida bo‘lib o‘tadi (Manzil: 100125, Toshkent shahri, Do‘rmon yo‘li ko‘chasi, 33-uy. Tel./Faks: (+99871) 262-31-63, faks: (+99871) 262-32-54, e-mail: info@iplt.uz, Ion-plazma va lazer texnologiyalari instituti majlislar zali).

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X.B. Ashurov
Ilmiy darajalar beruvchi Ilmiy kengash raisi, t.f.d., professor

I.D. Yadgarov
Ilmiy darajalar beruvchi Ilmiy kengash ilmiy kotibi, f.-m.f.d., professor

U.K. Maxmanov
Ilmiy darajalar beruvchi ilmiy kengash qoshidagi ilmiy seminar raisi, f.-m.f.d., professor.

KIRISH (falsafa doktori (PhD) dissertatsiya annotatsiyasi)

Dissertatsiya mavzusining dolzarbligi va ahamiyati. Bugungi kunda zamonaviy materialshunoslik va nanotexnologiyalarda fizik xususiyatlari boshqariladigan nanoo‘lchamli materiallarni sintez qilish va ularni amaliy qurilmalarga integratsiyalashga katta e’tibor qaratilmoqda. Istiqbolli materiallardan biri bo‘lgan uglerod nanonaychalari (UNN) yuqori mexanik mustahkamlik, elektr va issiqlik o‘tkazuvchanlik, kimyoviy barqarorlik hamda katta solishtirma sirt maydoniga ega bo‘lib, sensorlar, energiya saqlash qurilmalari, o‘tkazuvchi kompozitlar va boshqa funksional tizimlarda keng qo‘llanilishi mumkin. Biroq UNNlarning amaliy xususiyatlarini ta’minlash uchun ularning diametri, sirt zichligi, o‘sish yo‘nalishi va strukturaviy sifatini boshqarish talab etiladi.

Bug‘ fazadan kimyoviy cho‘ktirish (CVD) usulida UNNlarning nukleatsiyasi va o‘sishi katalizator nanozarrachalarining o‘lchami, zichligi va barqarorligiga bevosita bog‘liq. Fe katalizatori uglevodorodlarni parchalash va uglerodni eritish xususiyati tufayli UNN sintezida samarali bo‘lsada, yuqori haroratlarda zarrachalarning yiriklashishi, koalesensiyasi, taglik bo‘ylab diffuziyalanishi va uglerod bilan qoplanishi katalitik faollikning pasayishiga olib kelishi mumkin. Shu sababli yaxlit Fe yupqa plyonkasining termik ta’sir natijasida nanozarrachalarga ajralish qonuniyatlarini aniqlash boshqariladigan UNN sintezining muhim ilmiy masalasidir.

Fe nanozarrachalarining shakllanishi plyonka qalinligi va termik ishlov berish bilan birga katalizator joylashgan sirtning fizik-kimyoviy tabiatiga ham bog‘liq. SiO_2, Al_2O_3 va TiO_2 kabi bufer qatlamlar Fe atomlarining sirt bo‘ylab diffuziyasini cheklab, zarrachalarning o‘lchami, shakli va termik barqarorligiga ta’sir ko‘rsatadi. Bunda bufer qatlamning qalinligi, tuzilishi va Fe bilan o‘zaro ta’siri muhim omillar hisoblanadi. Biroq Fe plyonkasi qalinligi, termik ishlov parametrlari va bufer qatlam tabiatining birgalikdagi ta’sirini hamda ularning UNN morfologiyasi va strukturaviy sifatiga bog‘liqligini miqdoriy baholash masalasi yetarlicha o‘rganilmagan. Shu nuqtai nazardan, elektron-nurli fizik bug‘ cho‘ktirish (PVD) usulida hosil qilingan Fe yupqa qatlamlarining SiO_2, Al_2O_3, TiO_2 va sapfir sirtlarida nanozarrachalarga transformatsiyalanishini tadqiq etish, ularning o‘lchami va sirt zichligini boshqarish qonuniyatlarini aniqlash hamda UNN nukleatsiyasi va o‘sishiga ta’sirini baholash dolzarb ilmiy vazifa hisoblanadi. Dissertatsiyada Fe qatlamining qalinligi, termik ishlov rejimi va bufer qatlam turi nanozarrachalar morfologiyasini belgilovchi o‘zaro bog‘liq omillar sifatida tadqiq etilgan.

O‘zbekistonda ilm-fan, innovatsion texnologiyalar va zamonaviy materiallarni rivojlantirishga alohida e’tibor qaratilmoqda. Ilg‘or texnologiyalarni iqtisodiyotga joriy etish, ilmiy ishlanmalarni amaliyot bilan integratsiyalash va materialshunoslik tadqiqotlarini rivojlantirish ustuvor vazifalardan hisoblanadi. Mazkur dissertatsiya tadqiqoti O‘zbekiston Respublikasi Prezidentining 2022-yil 6-iyuldagi PF-165-son Farmoni[1], 2025-yil 27-dekabrdagi PQ-390-son qarori[2], 2026-yil 16-fevraldagi PF-

[1] O‘zbekiston Respublikasi Prezidentining Farmoni, 22.10.2024 yildagi PF-165-son
[2] O‘zbekiston Respublikasi Prezidentining qarori, 27.12.2025 yildagi PQ-390-son

21-son Farmoni[3] hamda sohaga oid boshqa me’yoriy-huquqiy hujjatlarda belgilangan vazifalarni amalga oshirishga muayyan darajada xizmat qiladi.

Tadqiqotning respublika fan va texnologiyalari rivojlanishining ustuvor yo‘nalishlariga mosligi. Mazkur dissertatsiya tadqiqoti O‘zbekiston Respublikasida fan va texnologiyalarni rivojlantirishning “Fizika, astronomiya, energetika va mashinasozlik” ustuvor yo‘nalishiga muvofiq bajarilgan.

Muammoning o‘rganilganlik darajasi.

Uglerod nanonaychalari sintezida bug‘ fazadan kimyoviy cho‘ktirish usulining samaradorligi va nanonaychalar parametrlarini boshqarish bevosita katalizator nanozarrachalari hamda taglik (bufer) qatlamining o‘zaro ta’siriga bog‘liqligi xalqaro va mahalliy ilmiy jamiyatlar tomonidan keng tadqiq etib kelinmoqda. XXI asrning boshlarida e’tirof etilgan klassik ishlarda (Z.P. Huang, J. Robertson) Ni, Co va Fe kabi o‘tish metallarining yupqa qatlamlaridan termik ishlov berish orqali nanozarrachalar hosil qilish hamda TiN, SiO_2 kabi tagliklarda ularning sirt zichligini oshirish imkoniyatlari ko‘rib chiqilgan. Keyinchalik Suven Li (2017) va boshqa tadqiqotchilar tomonidan ko‘p qatlamli katalizator modellarini qo‘llash orqali nanozarrachalar o‘lcham taqsimotini nazorat qilish mumkinligi ko‘rsatib berildi.

So‘nggi yillardagi xalqaro ilmiy izlanishlar metall katalizator qatlamining termik parchalnish jarayonida bufer qatlamlarining (Al_2O_3, TiO_2) hal qiluvchi rolini aniqlashga qaratilgan. Ta’kidlash joizki, Al_2O_3 bufer qatlami metall atomlarining sirt bo‘ylab diffuziyasini va ichki qatlamlarga yutilishini cheklashi, shuningdek, yuqori haroratlarda Ostwald yetilishi (Ostwald ripening) tufayli nanozarrachalarning aglomeratsiyalanishini kamaytirishi aniqlangan. Turli tadqiqot guruhlari (EB-PVD, atom qatlamlab cho`ktirish (ALD) va magnetron changlatish usullarini qo‘llagan holda) Fe katalizatori va Al_2O_3/SiO_2 tandem bufer qatlamlari kombinatsiyasi bir devorli uglerod nanonaychasi (BDUNN) hamda vertikal yo‘nalgan ko‘p devorli (VYUNN) nanonaychalar diametrini va zichligini samarali boshqarish imkonini berishini tasdiqlaganlar. Shunga qaramay, yupqa Fe metall qatlamlarining (sub-nanometr va nanometr qalinlikdagi) harorat va vaqt rejimlariga bog‘liq ravishda faza o‘zgarishlari hamda bufer qatlam interfeysida nanozarrachalarga transformatsiyalanishining miqdoriy qonuniyatlari hamon to‘liq o‘z yechimini topmagan. O‘zbekistonda UNNlar va nanostrukturalarni olish, ularning nazariy hamda eksperimental asoslarini o‘rganish bo‘yicha sezilarli ilmiy maktab shakllangan. Qarshi davlat texnika universitetida prof. A.A. Rahmonqulov rahbarligida atsetilen pirolizi asosida UNN sintezi bo‘yicha tajribalar o‘tkazilgan bo‘lsa, O‘zFA U.A. Arifov nomidagi Ion-plazma va lazer texnologiyalari institutida (IPLTI) bu boradagi tadqiqotlar tizimli yo‘lga qo‘yilgan. Xususan, U. Xalilov va uning ilmiy guruhi tomonidan molekulyar-dinamik modellashtirish usuli orqali UNN o‘sish va nukleatsiya jarayonlarining kompyuter simulyatsiyalari muvaffaqiyatli amalga oshirildi. Shu bilan birga, Institutning “Amaliy nanotexnologiyalar” laboratoriyasida nanokatalizator zarrachalari va yupqa qatlamli strukturalarni PVD hamda CVD usullarida olish, ularning morfologiyasini tahlil qilish hamda muqobil energiya manbalarida (akkumulyator anodlarida) qo‘llash

[3]O‘zbekiston Respublikasi Prezidentining Farmoni, 16.02.2026 yildagi PF-21-son

bo‘yicha amaliy va amaliy-innovatsion izlanishlar olib borilmoqda. Biroq, aynan elektron-nurli PVD usulida yotqizilgan Fe yupqa qatlamlarining SiO_2, Al_2O_3, TiO_2 va sapfir sirtlarida atomar darajadagi shakllanishi hamda bufer-katalizator interfeysidagi jarayonlarning UNN struktural sifatiga ta’sirini yaxlit tadqiq etish bo‘yicha izlanishlarni yanada chuqurlashtirish talab etiladi.

Dissertatsiya tadqiqotining dissertatsiya bajarilgan ilmiy tadqiqot muassasasining ilmiy tadqiqot ishlari bilan bog’liqligi. Tadqiqot Ion-plazma va lazer texnologiyalari institutida huzuridagi IL-5421101842-son "Nanokatalizatorlar: shakllanishi, strukturasi, o'lcham effektlari va ularning uglerod nanomateriallari sintezi jarayonlariga ta'siri" nomli fundamental loyihasi doirasida olib borilgan.

Tadqiqotning maqsadi. Elektron-nurli PVD usulida cho`ktirilgan Fe yupqa qatlamining qalinligi, termik ishlov rejimlari va bufer qatlam turining katalizator nanozarrachalari o`lchami va zichligiga hamda uglerod nanonaychalarining nukleatsiyasi, o’sishi va strukturaviy sifatiga ta`sir qonuniyatlarini aniqlash, shu asosda bir devorli nanonaychalar sintezi uchun optimal katalizator–bufer qatlam tizimi va sintez rejimlarini ishlab chiqish.

Tadqiqot vazifalari:

elektron-nurli PVD usulida SiO_2, Al_2O_3 va TiO_2 bufer qatlamlari sirtida cho`ktirilgan Fe yupqa qatlamining qalinligini bug`lanish vaqti va emissiya toki orqali boshqarish rejimlarini ishlab chiqish hamda qatlamning element va fazaviy tarkibini aniqlash;

uzluksiz Fe yupqa qatlami termik ishlov berish orqali diskret nanozarralar tizimiga o`tish bosqichari va chegaraviy shartlarini aniqlash;

SiO_2, Al_2O_3 va TiO_2 nanostrukturalarda hosil qilingan Fe yupqa qatlami qalinligi, termik ishlov harorati va davomiyligini nanozarrachalar o'lchami, sirt zichligi va o'lcham taqsimotiga ta`sirini miqdoriy baholash, zarracha diametri va plyonka qalinligi orasidagi empirik bog'lanishni aniqlash;

UNN nukleatsiyasi va o'sishining katalizator zarrachalari bilan bog'liqligini, jumladan o'sish jarayonlari va katalizator deaktivatsiyasi fizik-kimyoviy sabablarini o'rganish;

turli katalitik tizimlarda ($Fe/SiO_2/Si$, $Fe/Al_2O_3/SiO_2/Si$, $Fe/TiO_2/SiO_2/Si$, Fe/sapfir) sintez qilingan UNNlarning strukturaviy sifati va morfologiyasini solishtirib, optimal katalizator–bufer qatlam dizaynini aniqlash;

harorat va gaz sarfi parametrlariga bog‘liq holda CVD jarayonida bir devorli uglerod nanonaychalari nukleatsiya kinetikasiga bufer qatlamining ta’sirini tadqiq qilish.

Tadqiqotning ob’ekti. Tadqiqotning asosiy obyektlari sifatida SiO_2, Al_2O_3, TiO_2 bufer qatlamlar hamda sapfir tagligi sirtida sintez qilingan Fe nanozarralari va ular asosida shakllangan UNNlar o`rganilgan.

Tadqiqotning predmeti. Turli oksid bufer qatlamlar sirtida Fe nanozarralarining shakllanish jarayonlari, zarralar o`lchami va taqsimotiga ta`sir etuvchi termik ishlov berish parametrlarini optimallashtirish, katalizatorlar sirtida UNNlar nukleatsiya jarayonlari va zaharlanish sabablarini aniqlash, bir devorli UNNlar sintezi jarayonlari tadqiqot ishining asosiy predmeti hisoblanadi.

Tadqiqotning usullari. Tadqiqot ishini bajarish uchun namunalar sirtida SiO_2 qatlami hosil qilishda quruq termik oksidlanish metodi, Al_2O_3 va Fe yupqa qatlami hosil qilishda esa elektron nurli fizik bug` cho`ktirish usuli, TiO_2 qatlami hosil qilishda atom qatlamlab cho`ktirish, UNN sintezi uchun esa kimyoviy bug` cho`ktrish usulidan foydalanildi. Olingan namunalarning strukturaviy va morfologik tabiatini aniqlash uchun AFM, XPS, XRD, TEM, SEM, va Raman spektroskopiyasi singari analitik qurilmalardan foydalanildi.

Tadqiqotning ilmiy yangiligi:

ilk bor, Amorf SiO_2 va Al_2O_3 sirtlarida yuqori zichlikdagi (28 nm, 302 zarra/μm^2) kvazi-sferik zarralar, kristall sapfir va TiO_2 sirtlarida esa qirralangangan va nisbatan siyrak zarrachalar (mos ravishda ≈68 nm, 17 zarra/μm^2 va ≈56 nm, 87 zarra/μm^2) hosil bo'lishi ko`rsatildi. Shu bilan birga, zarralarning sirtda notekis taqsimoti va yiriklashishi orqali ushbu kristall tagliklarda Fe atomlarining sirt diffuziyasini cheklashi amorf Al_2O_3 ga nisbatan sustroq ekanligi isbotlandi;

Al_2O_3 (120 nm)/ SiO_2 (160 nm)/Si tizimida elektron-nurli PVD usulida cho'ktirilgan Fe yupqa qatlamini 700 °C da 5 daqiqa termik ishlov berishda hosil bo'ladigan nanozarrachalar o'rtacha diametri (d) va boshlang'ich qatlam qalinligi (h) orasida $d \approx k \cdot h$ bog'lanishi aniqlandi. Adabiyotda Fe asosli tizimlar uchun keltirilgan keng oraliq ($k \approx 2–10$) ushbu sharoit uchun **ilk bor** $k \approx 4–6$ gacha toraytirildi. Bu katalizator zarrachalari o'lchamini boshlang'ich qatlam qalinligi orqali oldindan baholash va boshqarish imkonini beradi;

ilk bor, SiO_2, Al_2O_3/SiO_2, TiO_2/SiO_2 bufer qatlamlar va sapfir tagliklarda sintez qilingan UNNlar kompleks tahlil qilinib, bunda Al_2O_3(120 nm)/SiO_2(160 nm) tandemida sintez qilingan UNNlar sifati bo`yicha $TiO_2$ ga nisbatan 19,2 %, tabiiy $SiO_2$ ga nisbatan 33,3 %, sapfirga nisbatan 41,7 % ustunligigi ko`rsatildi;

ilk bor, nisbatan qalin (8-9 nm) *Fe* qatlamlarida ham Al_2O_3*(120 nm)/*SiO_2*(160 nm)* strukturasi yuqori sirt g`adir-budurligi (*Ra=1,06 nm*) hamda Fe zarralari bilan nisbatan kuchli o`zaro ta`sirlashuvi sababli, 700 °C haroratda bir devorli UNN sintezida eng samarali buffer qatlam tandemi ekanligi nazariy hisoblashlar (Raman RBM, *d=0,9 - 1,1 nm*) va amaliy usullar (SEM tasvirlarida, *d=3,6 nm*) orqali isbotlandi.

Tadqiqotning amaliy natijalari. Ushbu tadqiqot natijalari turli tagliklar sirtida turli oksid bufer qatlamlardan foydalanib fizik va kimyoviy bug` cho`ktirish usullari orqali 300 zarra/μm^2 zichlikdagi o`rtacha diametri 25-30 nm bo`lgan Fe nanozarralari hosil qilishning optimal rejimlarini taqdim etadi. Bundan tashqari, katalitik hamda bufer qatlamlar qalinligi, termik ishlov harorati va vaqtini nazorat qilish orqali hosil bo`layotgan Fe zarralarini o`lchamini boshqarish imkoniyatlari aniqlangan. Ushbu zarralarni katalizator sifatida metan, vodorod gazlarini parchalashda, ammiak, Uglerod nanotolalari, Bor nitrit sintezida, fotoelektrokimyoviy suvni parchalash singari jarayonlarda faol qo`llanilishi mumkin. Shuningdek, ushbu tadqiqot ishida Fe nanokatalizatorlari yordamida vertikal yo`nalgan, bir devorli hamda ko`p devorli UNNlar sintez sharoitlari tajribalar orqali tasdiqlangan. UNNlar sifati va o`sish balandligini oshirish uchun yangi katalitik dizayn hosil qilish parametrlari ishlab chiqilgan.

Tadqiqot natijalarining ishonchliligi quyidagilar ta`minlaydi: zamonaviy standartlashtirilgan usullarning qo'llanilishi, yuqori aniqlikdagi asbob-uskunalardan foydalanilganligi, statistik tahlilga yetarli bo'lgan eksperimental ma'lumotlar hajmi va natijalarning qayta takrorlanganda barqaror saqlanishi.

Tadqiqot natijalarining ilmiy ahamiyati shundan iboratki, elektron-nurli PVD usulida cho'ktirilgan Fe yupqa qatlamlarining SiO_2, Al_2O_3, TiO_2 bufer qatlamlari va sapfir taglik sirtida nanozarrachalarga transformatsiyalanish qonuniyatlari hamda ularning o'lchami, shakli va sirt zichligiga ta'sir etuvchi omillar (qatlam qalinligi, termik ishlov harorati va davomiyligi, taglik tabiati) miqdoriy aniqlanib, katalizator zarrachalari o'lchamini boshqarishning ilmiy asoslari ishlab chiqildi. Shuningdek, katalizator–bufer qatlam interfeysi parametrlarining UNN nukleatsiyasi, o'sishi va strukturaviy sifatiga ta'siri ko'rsatilib, yuqori haroratda (850 °C) katalizator faolligini saqlovchi Al_2O_3/SiO_2 tandem bufer qatlami eksperimental asoslandi.

Tadqiqot natijalarining amaliy ahamiyati esa Fe katalizator qatlami qalinligini bug'latish vaqti va emissiya toki orqali aniq boshqarish rejimlari hamda yuqori zichlikdagi, bir jinsli Fe nanozarrachalarini hosil qilishning optimal termik ishlov sharoitlari aniqlanganligi bilan belgilanadi. Fe (8–9 nm)/ Al_2O_3 (120 nm) / SiO_2 (160 nm) / Si strukturasi asosida strukturaviy sifati yuqori (I_D/I_G=0,42), shu jumladan bir devorli (diametri 0,93–1,43 nm) UNNlar sintez qilish imkoniyati tajriba orqali ko'rsatildi. Olingan natijalar UNN asosidagi energiya saqlash tizimlari, o'tkazuvchan kompozit materiallar va sensorlar uchun katalizator–bufer qatlam parametrlarini maqsadli tanlash bo'yicha amaliy tavsiyalar sifatida xizmat qiladi.

Tadqiqot natijalarini joriy qilish.

Disertatsiya tadqiqoti davomida olingan natijalar O`zbekiston Respublikasi Fanlar akademiyasi Umumiy va noorganik kimyo institutida O’zbekiston–Turkmaniston ilmiy hamkorligi asosida 2024–2025-yillarda bajarilgan AL-7623031231 raqamli «Siklopentadienil temir dikarbonil dimer asosida uglerodli nanonaylar sintez qilish va ularni fizik-kimyoviy xususiyatlarini o’rganish» mavzusidagi fundamental loyihada bufer qatlamlar sirtida Fe nanozarralarini hosil qilishning aniqlangan maqbul kinetik sharoitlarida foydalanildi (Fe qatlam qalinligi 8–9 nm, 700 °C, 5 daqiqa; Al_2O_3 (120 nm)/SiO_2 (160 nm) tizimida o’rtacha diametri 25–30 nm, zichligi ≈300 zarra/μm^2) (O`zR FAning 2026-yil 4-noyabrdagi № 4/1255-3131 sonli ma’lumotnomasi). Bu natijalar katalizator zarralari o’lchamini nazorat qilish muammosini hal etdi: o’lchamlari qat’iy boshqariladigan faol katalitik markazlar asosida yuqori sifatli, mukammal kristallangan bir devorli uglerod nanonaychalarini sintez qilish va ularning xususiyatlarini optimallashtirish imkoni yaratildi.

«Armatura-Izolyator zavodi (AIZ)» MChJ qo’shma korxonasida 2024–2026-yillarda amalga oshirilgan «Kremniyorganik polimer asosli IPP-10/1000-8A UHL1 va LK 70/10-3 UXL1 yuqori kuchlanish izolyatorlar samaradorligini oshirish» nomli amaliy loyihada Fe/Al_2O_3/SiO_2/Si, Fe/TiO_2/SiO_2/Si va Fe/SiO_2/Si geterotuzilmalari sirtida CVD usulida nanonaychalar sintezi bo’yicha olingan qonuniyatlardan, ya’ni oraliq qatlam tabiati, harorat rejimi va reaksiya

davomiyligining nanotuzilmalar morfologiyasi hamda sifatiga ta’siridan foydalanildi («AIZ» MChJ QKning 2026-yil 16-yanvardagi 19-sonli ma’lumotnomasi). Bu natijalar izolyatorlar kompozitida nanonaychalarning optimal konsentratsiyasi va bir tekis dispersiyalanishini ta’minlash muammosini hal etdi, buning natijasida IPP-10/1000-8A va LK 70/10-3 rusumli izolyatorlarning tashqi muhit omillari va mexanik kuchlanishlarga nisbatan ekspluatatsion bardoshliligi oshdi.

Tadqiqot natijalarini aprobatsiya qilish. Tadqiqotning asosiy natijalari 3 ta respublika miqyosidagi hamda 4 ta xalqaro konferensiyalarda muhokama qilingan..

Tadqiqot natijalarining e’lon qilinganligi. Dissertatsiya mavzusining asosiy qismini yoritib beruvchi natijalar asosida jami 12 ta ilmiy ishlar nashr etilgan bo`lib, ulardan 2 tasi xalqaro jurnallarda va 3 tasi mahalliy OAK doktorlik disertatsiyasining asosiy ilmiy natijalarini chop etish tavsiya etiladigan jurnallarda nashr ettirilgan.

Dissertatsiyaning tuzilishi va hajmi. Dissertatsiya ishi tuzilish jihatidan kirish, 4 bob, xulosalar qismi, adabiyotlar ro`yxati va ilovalardan tashkil topgan. Uning asosiy qismi 117 bet matn, 56 ta rasm va 7 ta jadvaldan iborat.

DISSERTATSIYANING ASOSIY MAZMUNI

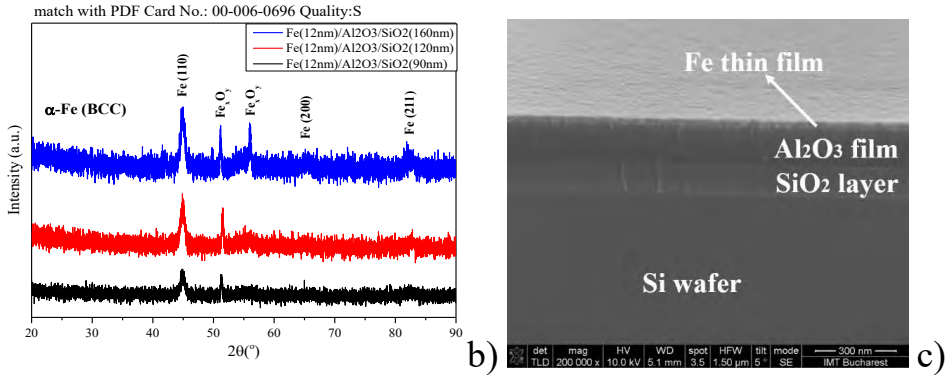
Kirish qismida katalizator–bufer qatlam interfeysida Fe nanozarrachalarining shakllanishi va uning uglerod nanonaychalari o'sishiga ta'siri mavzusining dolzarbligi ko'rsatilib, tadqiqotning maqsadi, vazifalari, ob'ekti, predmeti va qo'llanilgan usullari bayon etilgan. Ishning respublika fan va texnologiyalarini rivojlantirishning ustuvor yo'nalishlariga mosligi keltirilgan. Shuningdek, olingan natijalarning ilmiy yangiligi, ilmiy va amaliy ahamiyati, ishonchliligi, aprobatsiyasi, e'lon qilinganligi hamda dissertatsiyaning tuzilishi va hajmi haqida ma'lumot berilgan.

Dissertatsiyaning birinchi bobi "Adabiyotlar tahlili va ilmiy muammolar holati" deb nomlangan bo'lib, unda uglerod asosli nanomateriallarning tuzilishi, xossalari va o'sish mexanizmlari, xususan 0D, 1D, 2D va 3D uglerod nanostrukturalari, ularning xirallik turlari va amaliy qo'llanish sohalari bo'yicha ilmiy manbalar tahlil qilingan. Bobda UNN sintezining asosiy usullari - yoy razryadi, lazer abelyatsiyasi va CVD usullari - qiyosiy tahlil qilinib, ularning afzallik va kamchiliklari ko'rsatib berildi. UNN o'sishida Fe, Ni va Co nanokatalizatorlarining roli, xususan Fe asosidagi katalizatorlarning morfologiyasi va faolligiga ta'sir etuvchi omillar - zarracha o'lchami, taglik tabiati, termik ishlov parametrlari - batafsil yoritildi. Fe nanostrukturalarni olish usullari (PVD, ALD, magnetron changlatish) o'zaro solishtirilib, ularning texnologik imkoniyatlari tahlil qilindi. Bobning yakuniy qismida UNN sintezida CVD metodining harorat, vaqt, katalizator qalinligi kabi texnologik parametrlar bilan o'zaro bog'liqligi ilmiy adabiyotlar asosida umumlashtirildi hamda mavjud tadqiqotlardagi bo'shliqlar - xususan, elektron-nurli PVD usulida yotqizilgan Fe yupqa qatlamlarining turli oksid bufer qatlamlar sirtida atomar darajadagi shakllanish qonuniyatlarining yetarlicha o'rganilmaganligi asoslab berildi.

Ikkinchi bob "Bufer-katalizator qatlamlarini hosil qilishning optimal sharoitlari va uglerod nanonaychalar sintezi uchun tajriba-tadqiqot hamda tahlil usullari" deb nomlangan bo'lib, unda tadqiqotda qo'llanilgan boshlang'ich materiallar - monokristall kremniy (KES, (100) orientatsiyali, qalinligi 375 μm) va sapfir tagliklar hamda ularni tayyorlash jarayonlari tavsiflangan.

Bu bobda SiO_2 qatlamini quruq termik oksidlanish usulida hosil qilish va uning qalinligini spektroskopik ellipsometriya yordamida o'lchash, Si/SiO_2 struktura sirtida Al_2O_3 qatlamini elektron-nurli PVD usulida hosil qilish, TiO_2 bufer qatlamini atom qatlamlab o'stirish (ALD) usulida sintez qilish hamda Fe kataliz qatlamini elektron-nurli bug'latish orqali olish bosqichlari batafsil bayon qilingan. Shuningdek, uglerod nanonaychalarini CVD usulida sintez qilish texnologiyasi va optimal sharoitlari, hosil qilingan namunalarning strukturaviy va morfologik xususiyatlarini aniqlashda qo'llanilgan SEM, TEM, STEM, AFM, XRD, XPS, FTIR va Raman spektroskopiyasi kabi analitik tahlil usullarining ishlash prinsiplari va o'lchov sharoitlari yoritildi.

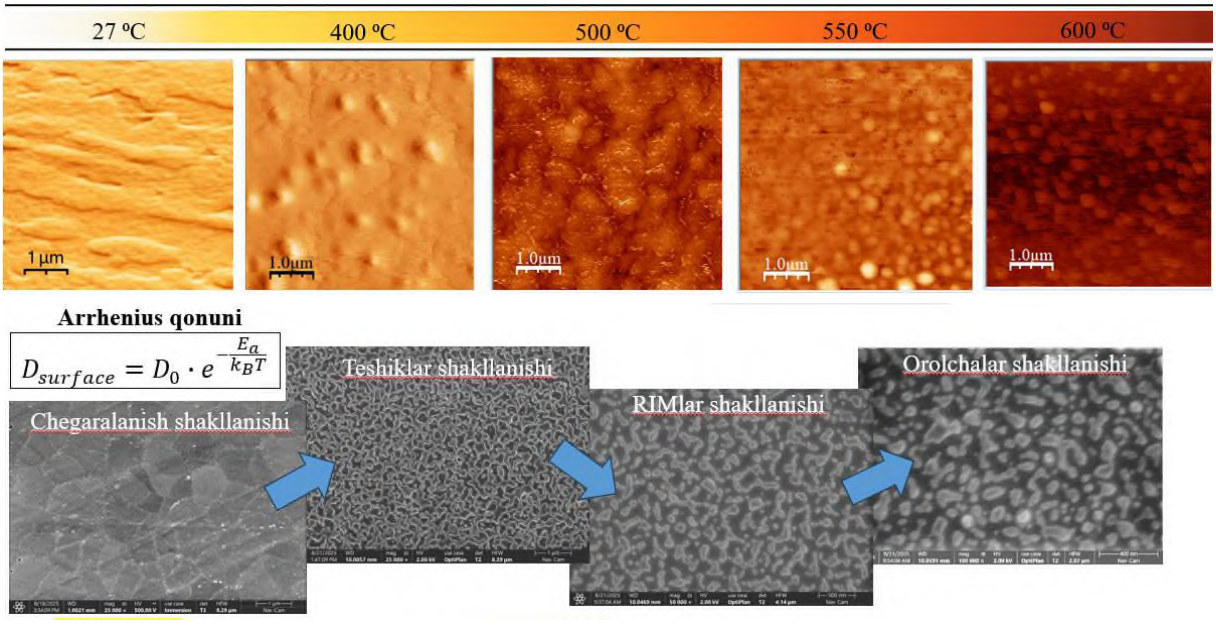
Uchinchi bob "Fe nanostrukturalarning shakllanishi va ularga ta'sir etuvchi omillar tahlili" deb nomlangan bo'lib, bunda bug' fazadan cho'ktirilgan Fe yupqa qatlamlarining Al_2O_3 va SiO_2 bufer qatlamlari ustida hosil bo'lgan fazaviy holati SEM, EDX, XPS va XRD usullari yordamida tahlil qilingan. XPS spektrlari Fe atomlarining asosan metall holatda mavjudligini va sirtida qisman oksidlanish ehtimoli borligini ko'rsatdi (1-rasm).



a) b) c)

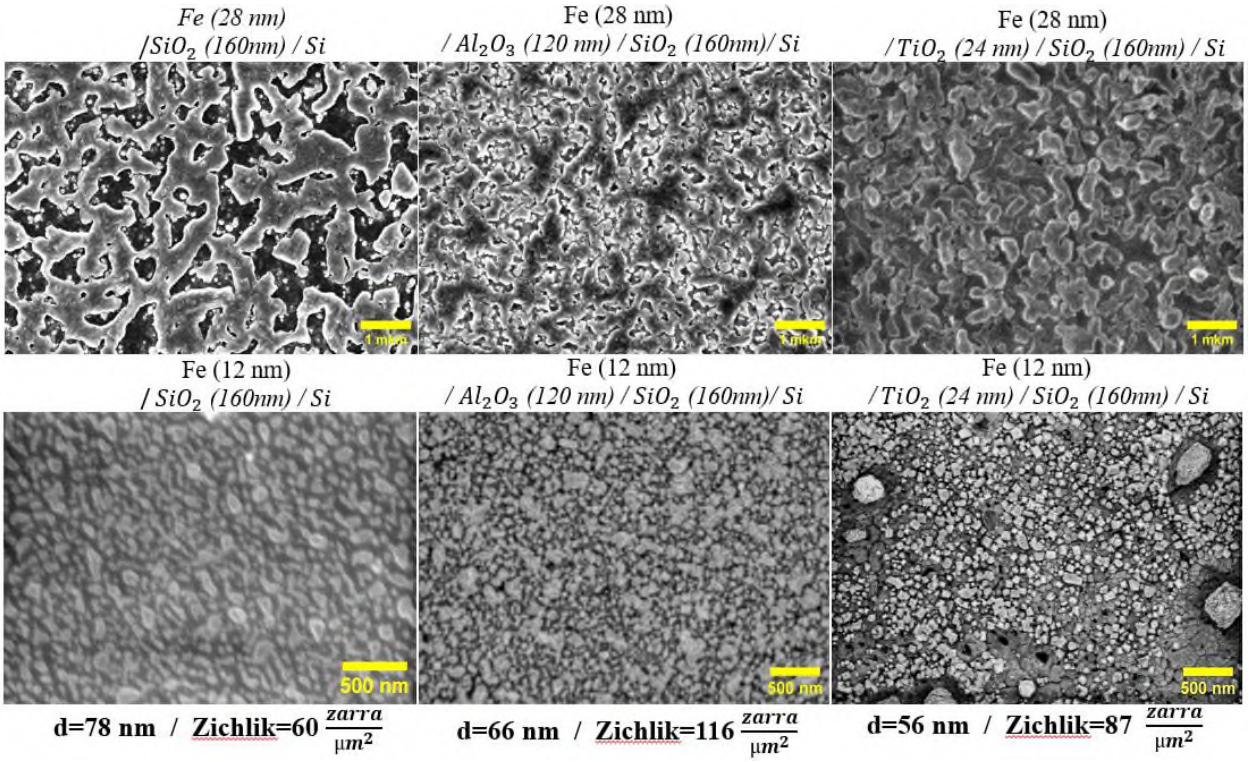
1-rasm. Yupqa Fe qatlamining XRD tahlillari (a), ko`ndalang kesim SEM tasviri (b) va XPS tahlili (c).

Termik ishlov ta'sirida kataliz yupqa qatlamidan nanostrukturalar shakllanish mexanizmi o'rganilib, 8 nm qalinlikdagi Fe qatlamida harorat oshishi bilan uzluksiz plyonkaning diskret nanozarrachalar tizimiga parchalanishi kuzatildi: 550 °C atrofida uzluksiz qatlam to'liq parchalanib, sferik shaklga yaqin nanozarrachalar hosil bo'lishi Atom kuch mikroskopi tasvirlari orqali ochib berildi (2-rasm). Bundan tashqari uzluksiz qatlamdan diskret nanozarralar shakllanish jarayonlarini yanada aniqroq tushunish uchun o`tkazilgan tadqiqotlarda, plyonka termik qizdirish natijasida dastlab atomlar tebranishi kuchayadi va energetik noqulay joylarda chegaralanish shakllana boshlaydi. Keyingi bosqichda, sirtda teshiklar hosil bo`ladi va ularning kattalashishi oqibatida Rimlar, ulardan keyin esa nanoorolchalar shakllanadi. Sirtda energiya qayta taqsimlanishi hamda sirt diffuziyasi tufayli atomlar ko`chishi sodir bo`ladi va bu jarayon Arrenius qonuni bo`yicha ifodalandi.



2-rasm. 8 nm qalinlikdagi uzluksiz Fe yupqa plyonkasining diskret nanozarralarga transformatsiya jarayoninig a) AFM va b) SEM tasvirlari.

Qatlam qalinligining nanostrukturalar shakllanishidagi ahamiyati, shuningdek termik ishlov berish harorati va vaqtining hosil bo'layotgan nanozarralar morfologiyasiga ta'siri SEM tahlillari keltirilgan: 28 nm qalinlikdagi Fe qatlamlari 700 ^{0}C haroratda 10 daqiqa termik ishlov jarayonida to'liq parchalanmay, notekis morfologiyali strukturalar hosil qilgani, 12 nm qalinlikdagi qatlamlarda esa ancha kichik va bir jinsli nanozarrachalar tizimi shakllangani aniqlandi (3-rasm).



3-rasm. Turli tagliklar va qalinliklardagi Fe qatlamining 700 ^{0}C da 10 daqiqa termik ishlov berilgandan keyingi sirt morfologiyalari SEM tasvirlari.

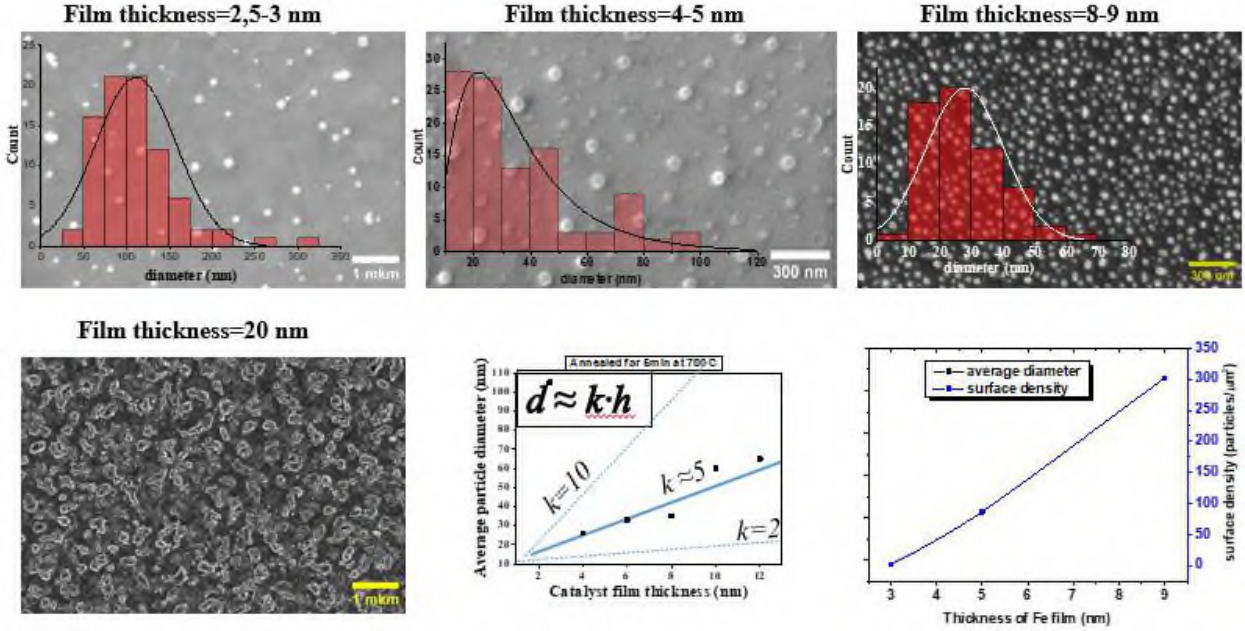
Bunda SiO_2 sirtida o`rtacha qiymati 78 nm, $Al_2O_3/SiO_2$ sirtida 66 nm va $TiO_2/SiO_2$ sirtida esa 56 nm o`lchamdagi nanozarralar aniqlandi. Nanozarralar

o`lchamini boshqarish maqsadida amalga oshirilgan tadqiqotlarda 3-20 nm qalinlikdagi Fe yupqa qatlamlari $SiO_2$/Si namuna sirtida EB-PVD usulida hosil qilindi. Olingan yupqa qatlamlarga 700 $^0$C da 5 min termik ishlov berilganda ulardan hosil bo`ladigan zarralar SEM orqali tahlil qilindi (4-rasm). 8-9 nm qalinlikdagi yupqa qatlamdan o`rtacha o`lchami 25-30 nm bo`lgan va zichligi 300 zarra/μm^2 nanozarralar shakllanishi aniqlandi.

Bundan tashqari nanozarralar diametri (d) va yupqa qatlam qalinligi (h) orasidagi o`zaro bog`liqlik (1) ifoda orqali hisoblandi:

$$d=k\cdot h \qquad (1)$$

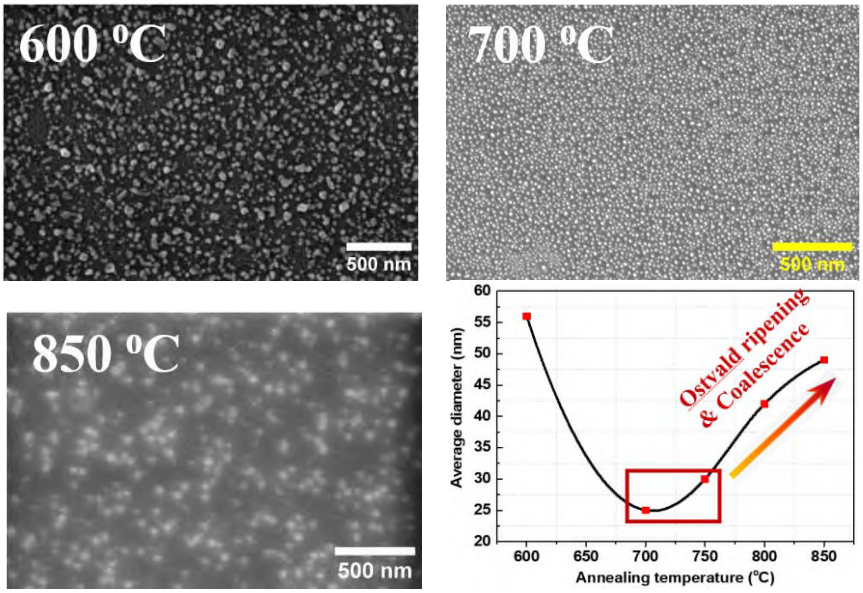
unga ko`ra, adabiyotlarda keltirilgan k koeffitsient o`zgarish oralig`i [2-10] dan [4-6] oraliqqacha toraytirildi. Bu tadqiqot kerakli o`lchamdagi nanozarralar sintezida yuqori aniqlikda ishlash imkoniyatini yanada oshiradi va zarralar o`lchamini qatlam qalinligi orqali boshqarish imkonini beradi.



4-rasm. Turli qalinlikdagi Fe yupqa qatlamlarining 700 ^{0}C da 5 min termik ishlovdan so`ng SiO_2/Si sirtida hosil qilgan zarralari morfologiyasi.

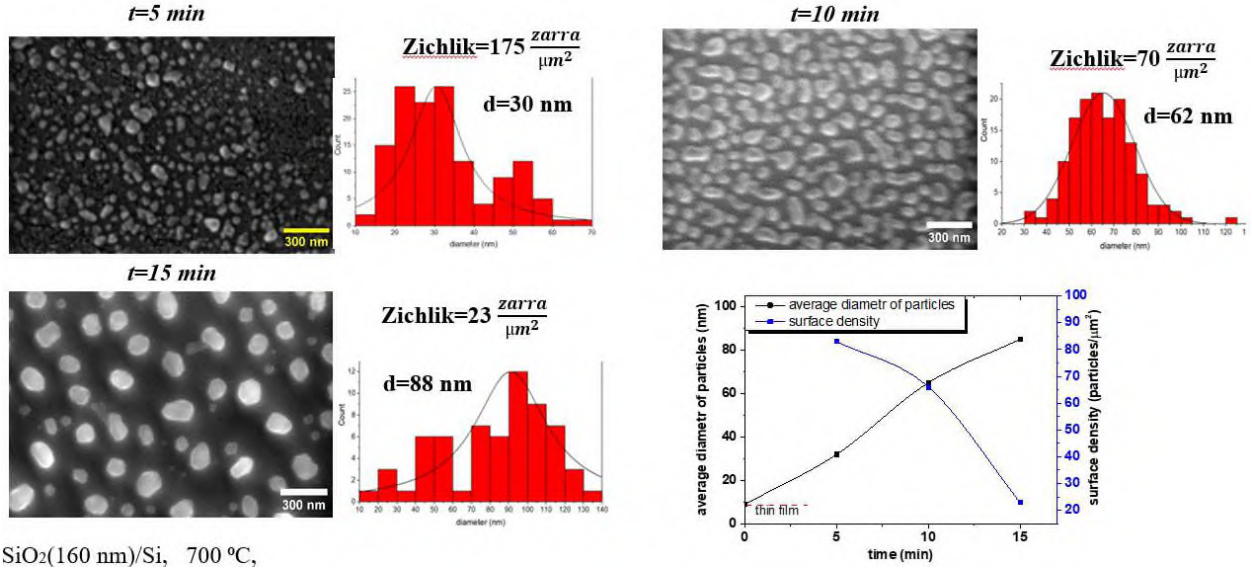
Nanozarralar o`lchamini boshqarishda termik ishlov haroratining ta`sirini aniqlash uchun turli haroratlarda olib borilgan tadqiqotlarda esa 700-750 ^{0}C harorat oralig`i eng kichik diametrli va eng yuqori zichlikdagi Fe nanozarralari shakllanishi uchun zarur muhitni yaratishi aniqlandi. 800 C va undan yuqori haroratlarda esa sirtda atomlar migratsiyasi, Osvald yetilishi va koalisensiya jarayonlari hisobiga zarralar o`lchamlari oshib ketishi tasdiqlandi.

Nanozarralar o`lchami nafaqat boshlang`ich qatlam qalinligi va termik ishlov haororatiga, balki ular termik ishlov davomiyligiga ham bog`liq va bu tadqiqot natijalari esa quyidagi xulosalarni berdi:



5-rasm. Turli haroratlarda termik ishlov berilgan 8-9 nm qalinlikdagi Fe yupqa qatlamidan hosil bo`lgan zarralar sirt morfologiyasi va ularning haroratga bog`liqlik grafigi.

Termik ishlov berish vaqti istalgancha davom etmasligi, va bunda ishlov vaqti haddan tashqari ko`p bo`lsa, nanozarralar qo`shni atomlar bilan qo`shilib ketish xavfini oshiradi. Tadqiqot natijalaridan ko`rinib turibdiki, 10 daqiqa davom etgan termik ishlovdan so`ng, zarralarning katta qismi koalisensiyaga uchragan va o`rtacha diametri 5 daqiqalik termik ishlovga qaraganda 2 baravar, zichligi esa 2 barvardan ko`proq kamayganligi aniqlandi. 15 daqiqalik termik ishlov natijalari esa 5 daqiqalik bilan solishtirilganda diamteri deyarli 3 barobar, zichligi 7 barobarga kamayib ketganligi aniqladi.

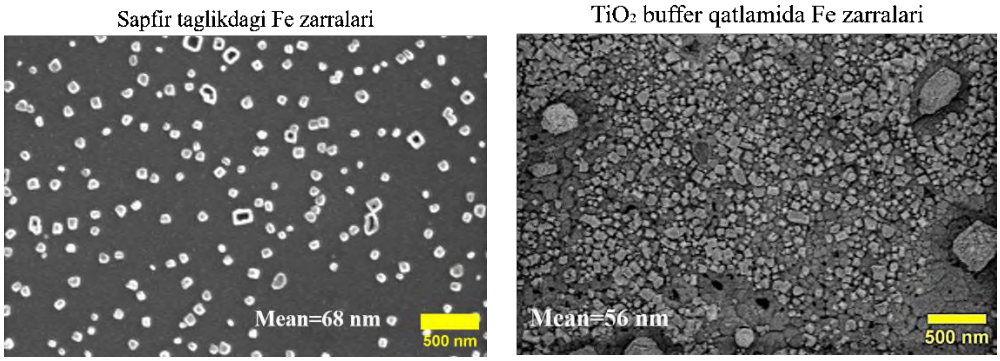


SiO_2(160 nm)/Si, 700 °C,

6-rasm. 700 C haroratda turli vaqt davomiyligida termik ishlov berilgan 8-9 nm qalinlikdagi Fe yupqa qatlamidan hosil bo`lgan zarralar SEM tasvirlari va sirt taqsimot grafiklari.

Yuqorida bayon qilingan amorf SiO_2 va Al_2O_3 qatlamlar sirtida hosil qilingan nanozarralar shakl jihatidan sferik va kvazi-sferik ekanligi SEM tasvirlaridan ma`lum.

Ammo kristall tagliklar sirtida Fe nanozarralari hosil qilish bo`yicha tadqiqotlar hozirgi kunda dunyo adabiyotlarida kam uchraydi. Sapfir va anataz fazadagi $TiO_2$ bufer qatlamlari sirtida 8-9 nm qalinlikdagi Fe yupqa qatlamiga termik ishlov berish natijasida hosil bo`lgan nanozarralar morfologiyasi shuni ko`rsatadiki (7-rasm), sapfir taglikda zarrachalar o`rtacha o`lchami 68 nm va zichligi 22 zarra/$\mu m^2$, $TiO_2$ da esa o`rtacha o`lchami 56 nm, zichligi 87 zarra/μm^2.



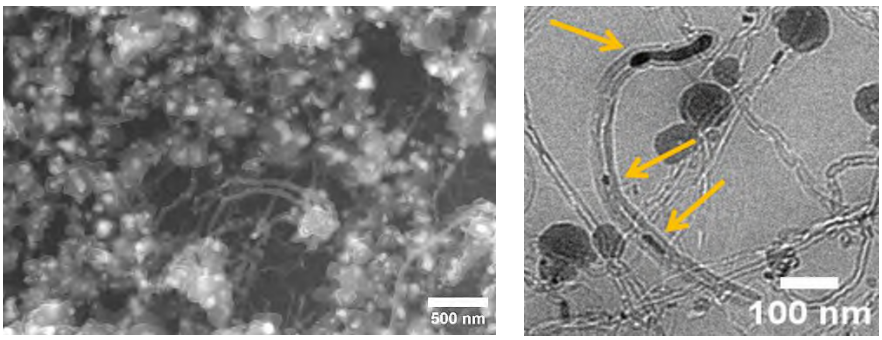
7-rasm. Sapfir va TiO_2 bufer qatlami sirtida hosil qilingan Fe nanozarralari.

Bu natijalar SiO_2 va Al_2O_3 bilan taqqoslaganda, TiO_2 bufer qatlami va sapfir taglikda atomlarning sirtdagi diffuziyasi yaxshi kechganligi va bu zarralar o`rtacha o`lchamining yiriklashishiga asosiy omil bo`lganligi bilan izohlandi. Bu yerda yana bir jihat, zarralarning shakli qirrali tuzilishga ega ekanligi bu esa taglikning kristallik xususiyati tufayli bo`lishi mumkinligi bilan izohlandi.

To‘rtinchi bob "Katalizator zarralari hamda bufer qatlami parametrlari va tabiatining UNN sinteziga ta'siri" deb nomlangan bo'lib, unda UNN nukleatsiyasi va o'sish mexanizmlarining katalizator zarrachalari bilan bog'liqligi tahlil qilingan: uglerod atomlarining katalizator sirtida adsorbsiyalanishi, zarracha ichiga diffuziyalanishi va kritik konsentratsiyaga yetgach grafitik faza ko'rinishida ajralib chiqishi jarayonlari o'rganildi. Ko'ndalang kesim SEM tasvirlarida katalizator zarrachalarining nanonaycha uchida joylashishi UNN o'sishi uchdan o'sish (tip-growth) mexanizmi asosida kechganini tasdiqladi.

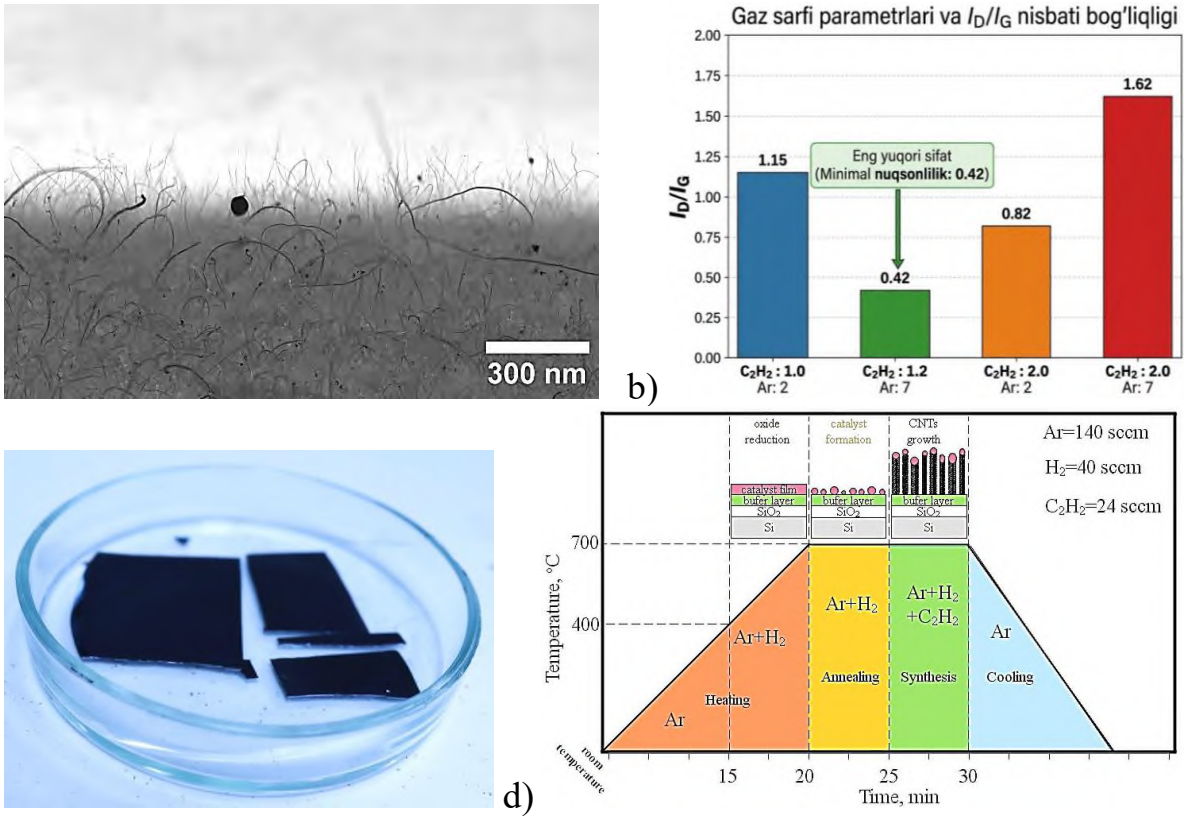
CVD usulida sintez qilingan (600 °C, 5 min, Ar/C_2H_2=2:2) namunaning SEM va TEM tahlillari uglerod nanonaychalari (UNN) o‘sishida katalizator zarrachalaridagi nukleatsiya jarayoni hal qiluvchi bosqich ekanligini ko‘rsatdi. Tajribada zarrachalarning faqat bir qismidangina ingichka UNNlar o‘sib chiqqan, asosiy qismi esa faol o‘sish markaziga aylanmagan (8a-rasm).

Bunga uglerod atomlarining zarrachaga kirish va ichki diffuziya tezliklari o‘rtasidagi muvozanatning buzilishi sabab bo‘lgan: uglerod zarracha ichiga diffuziyalanib to‘yinish darajasiga (supersaturatsiya) yetish o‘rniga uning sirtida to‘planib, zarrachani passivlovchi «yadro–qobiq» (core–shell) yoki nanopiyozcha strukturasini shakllantirgan (katalizator bloklanishi/zaharlanishi). Shuningdek, o‘sib borayotgan UNN devorlaridagi strukturaviy nuqsonlar katalizator bilan kuchli ta’sirlashib, uning aktiv massasining bir qismini yulib ketishi oqibatida o‘sish terminatsiyasi sodir bo‘lishi TEM orqali tasdiqlandi.



8-rasm. a) 600 ºC harorat va (Ar/C_2H_2=2:2) gaz oqimida sintez qilingan UNNlar SEM tasviri va b) 700 ºC harorat va (Ar/C_2H_2=7:2) gaz oqimida sintez qilingan UNNlar TEM tasviri

Ushbu eksperimental morfologik natijalar atomistik simulyatsiyalarda keltirilgan uglerod to‘planishi, supersaturatsiya va nukleatsiya mexanizmlari bilan to‘liq mos kelib, yuqori zichlikdagi sifatli UNN olish uchun diffuziya va sirt reaksiyalari kinetik muvozanatini aniq boshqarish zarurligini ko‘rsatadi.



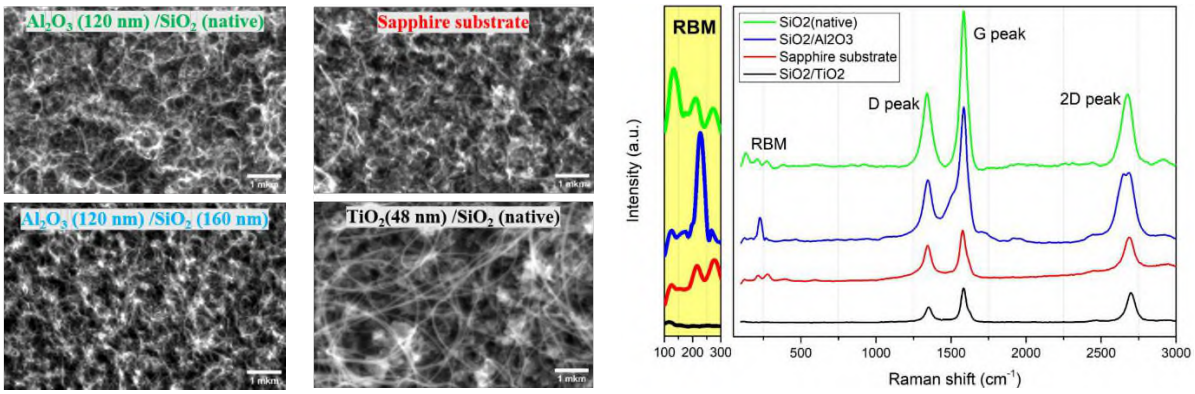
9-rasm. Uchdan o`sish mexanizmi asosida shakllangan UNN lar ko`ndalang kesim SEM tasviri (a) va nanonaychalar sifatining gaz sarfi parmetrlariga bog`liqligi grafigi (b), Si taglik sirtida sintez qilingan UNN real tasviri (c), CVD jarayonining vaqt bo`yicha xronologiyasi (d).

Eksperimental natijalar UNNlarning o‘sishi asosan VLS (*Vapor–Liquid–Solid*) va uchdan o‘sish (*tip-growth*) mexanizmlari orqali kechganini ko‘rsatdi. Ko‘ndalang kesim SEM tasvirlarida o‘sayotgan nanonaychalar uchida ularning diametriga teng yoki biroz yirikroq bo‘lgan katalizator zarrachalari (kontrastli nuqtalar) mavjudligi aniqlandi. Zarrachalarning substrat yuzasida qolmasdan (*base-growth* o‘rniga) nanonaycha uchi bilan yuqoriga ko‘tarilishi katalizator hamda substrat o‘rtasidagi

adgezion o‘zaro ta’sir kuchi nisbatan zaif ekanligidan dalolat beradi; natijada uglerodning yarim suyuqlangan zarracha ichida diffuziyalanishi va supersaturatsiyasi zarrachani taglikdan ajratib, uchdan o‘sish rejimini ta’minlagan.

Raman spektroskopiyasi tahlillari gaz komponentlari (C_2H_2:Ar) nisbati UNNning strukturaviy mukammalligiga sezilarli ta’sir ko‘rsatishini aniqladi. C_2H_2:Ar $= 1.2:7$ rejimida I_D/I_G nisbati minimal qiymatga (0.42) yetib, eng yuqori darajadagi kristallik va minimal nuqsonlilikka erishildi. Atsetilen sarfining ortishi (C_2H_2:Ar $= 2.0:7$) esa sirtda amorf uglerod to‘planishi hisobiga nuqsonlilik darajasini keskin oshirib yubordi ($I_D/I_G = 1.62$).

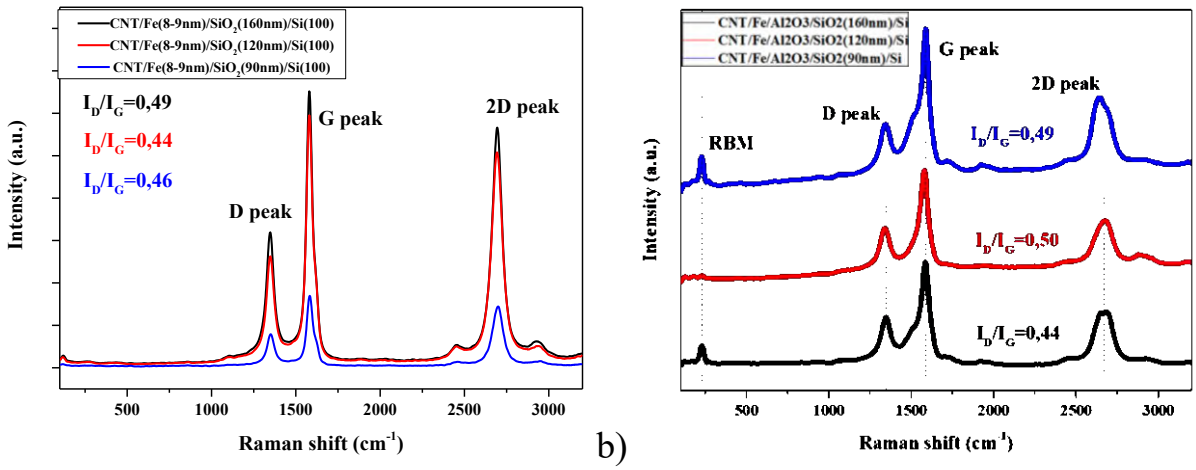
Katalizator morfologiyasi va hosil bo‘layotgan uglerod nanonaychalarining (UNN) strukturaviy sifatiga taglik hamda bufer qatlamining ta’siri SEM va Raman spektroskopiyasi yordamida qiyosiy o‘rganildi.



10-rasm. Turli tagliklarda sintez qilingan UNNlari sirt morfologiyasi (a) va Raman spektrlari (b).

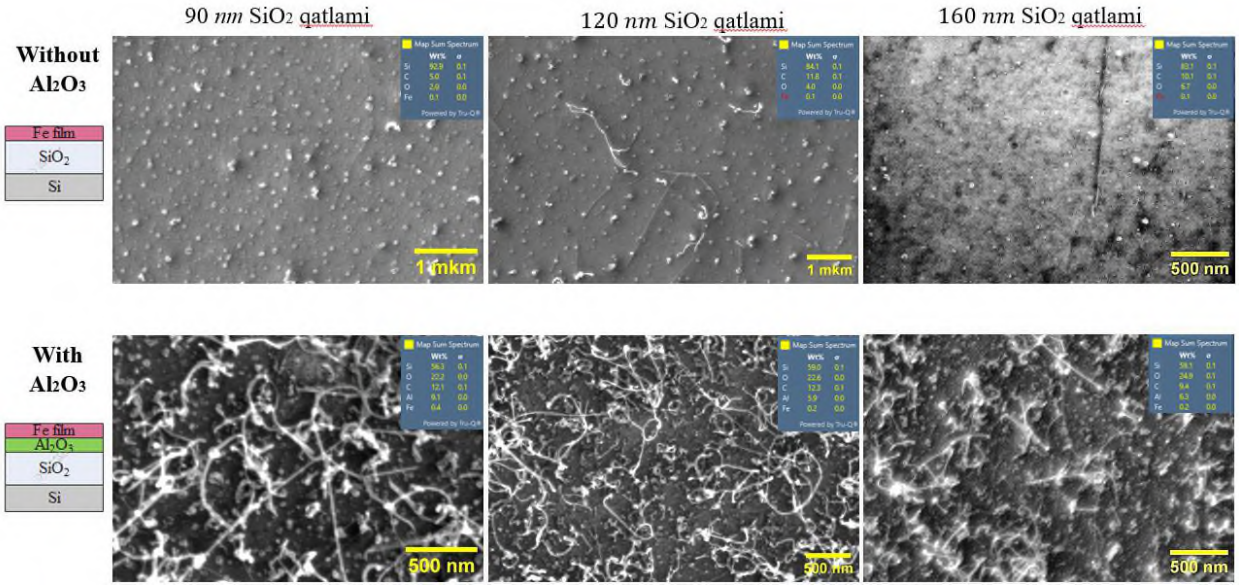
Tahlillar shuni ko‘rsatdiki, Fe (8–9 nm)/Al_2O_3(120 nm)/SiO_2 (160 nm)/Si geterostrukturasi eng optimal katalitik tizim hisoblanadi. Unda qalin SiO_2 va Al_2O_3bufer qatlamlari katalizator zarrachalarining aglomeratsiyalanishini samarali cheklab, ularning yuqori dispersiyali va bir tekis taqsimotini ta’minlagan; natijada eng yuqori zichlikdagi, past nuqsonlilikka ega ($I_D/I_G = 0.42$) bir devorli uglerod nanonaychalari (diametri 0.93–1.43 nm) olindi. Taqqoslash uchun, Fe (8–9 nm)/TiO_2 (48 nm)/SiO_2 (4 nm)/Si strukturasida hosil bo‘lgan zarrachalar qirrali morfologiyaga ega bo‘lib, koalesensiya to‘liq cheklanmagani sababli nanonaychalar diametri yirikroq, nuqsonlilik esa yuqoriroq ($I_D/I_G = 0.52$) bo‘ldi. Tabiiy oksidli Fe (8–9 nm)/Al_2O_3 (120 nm)/SiO_2 (4 nm)/Si strukturasida esa dispersiya yetarli nazorat qilinmagani oqibatida sifat ko‘rsatkichi pasaygan ($I_D/I_G = 0.63$, diametri 0.91–1.25 nm). Eng yuqori nuqsonlilik esa Fe/Sapfir tagligida kuzatilib ($I_D/I_G = 0.72$, diametri 0.90–1.20 nm), bu sapfir kristall panjarasi ta’sirida Fe zarrachalarining yirik va siyrak shakllanishi bilan izohlandi.

Shunday qilib, ko‘rib chiqilgan tizimlar orasida Fe/Al_2O_3/SiO_2/Si geterostrukturasi termik aglomeratsiyani bartaraf etish, nanozarrachalar o‘lchamini samarali boshqarish hamda yuqori sifatli bir devorli UNNlar sintez qilish uchun eng samarali asos ekanligi aniqlandi.



11-rasm. Turli qalinlikdagi SiO_2 va Al_2O_3/SiO_2 strukturalarda 700 °C haroratda sintez qilingan UNNlar Raman spektrlari.

CVD jarayonida (700 °C, 5 min) SiO_2 dielektrik qatlami qalinligining (90, 120 va 160 nm) nanonaychalar sifatiga ta'siri Raman spektroskopiyasi orqali baholandi. Barcha namunalarda sp^2-uglerod karkasiga xos bo'lgan asosiy Raman cho'qqilari ($D \approx 1350\ \text{cm}^{-1}$, $G \approx 1580\ \text{cm}^{-1}$ va $2D \approx 2700\ \text{cm}^{-1}$) qayd etilib, nuqsonlilik darajasi ko'rsatkichi tor diapazonda ($I_D/I_G \approx 0.44$–0.49) o'zgardi. Eng past nuqsonlilik 120 nm qalinlikdagi SiO_2 qatlamida ($I_D/I_G \approx 0.44$), nisbatan yuqorirog'i esa 160 nm qatlamda ($I_D/I_G \approx 0.49$) kuzatildi; biroq umumiy holda SiO_2 qalinligining o'zgarishi nanonaychalarning kristallik darajasiga keskin yoki tub ta'sir ko'rsatmasligi aniqlandi.

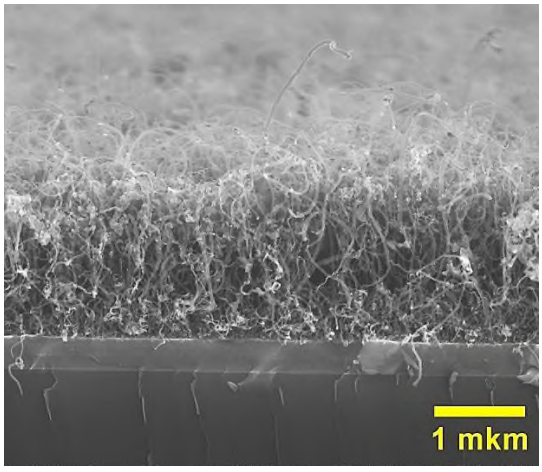


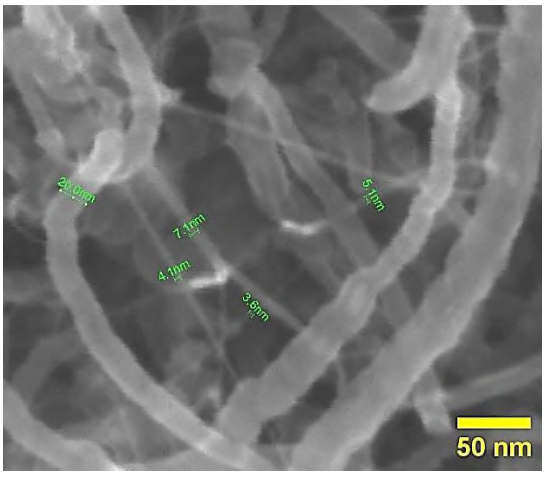
12-rasm. Turli qalinlikdagi SiO_2 va Al_2O_3/SiO_2 strukturalarda 850 °C haroratda sintez qilingan UNNlar sirt morfologiyasi.

Yuqori harorat sharoitida Fe nanozarrachalarining termik aglomeratsiyasini cheklash va ularning barqaror dispersiyasini ta'minlash maqsadida kiritilgan Fe (8–9 nm)/Al_2O_3 (120 nm)/SiO_2/Si ko'p qatlamli geterostrukturasi grafitlashish darajasini sezilarli darajada oshirdi ($I_D/I_G < 0.5$). Eng muhimi, past to'lqin sonlari sohasida (100–300 cm^{-1}) radial nafas olish modasining (RBM) paydo bo'lishi Al_2O_3 bufer qatlami yordamida nafaqat yuqori sifatli grafitik tartib, balki bir devorli uglerod nanonaychalari (BDUNN) hosil bo'lishi ta'minlanganini tasdiqladi.

Yuqori haroratli uglevodorodlar pirolizi sharoitida katalitik tizimning termik barqarorligini baholash maqsadida $Fe/SiO_2/Si$ va $Fe/Al_2O_3/SiO_2/Si$ strukturalarida 850 °C da uglerod nanonaychalari sintezi solishtirma tahlil qilindi. Tahlillar shuni ko‘rsatdiki, faqat SiO_2 bufer qatlamiga ega namunada nanonaychalar o‘sishi deyarli kuzatilmadi; bu yuqori haroratda Si atomlarining jadallashgan diffuziyasi hisobiga katalitik nofaol temir silisidlari (Fe–Si) fazalarining hosil bo‘lishi hamda Fe zarrachalarining kuchli koalesensiyaga uchrab passivlanishi bilan izohlandi.

Aksincha, oraliq Al_2O_3 bufer qatlami kiritilgan geterostrukturada 850 °C da ham zarrachalarning o‘lchami va faol dispersiyasi to‘liq saqlanib qolib, sirt bo‘ylab yuqori zichlikdagi, o‘zaro chalkashgan UNN massivlari o‘sishi ta’minlandi. Bu Al_2O_3 qatlamining silisidlanishga qarshi samarali diffuzion to‘siq vazifasini o‘tashi hamda termik aglomeratsiyani cheklashi bilan bog‘liq bo‘lib, ushbu ko‘p qatlamli tizimning keng harorat diapazonida yuqori strukturaviy va kimyoviy barqarorlikka ega ekanligini tasdiqlaydi.

a) 

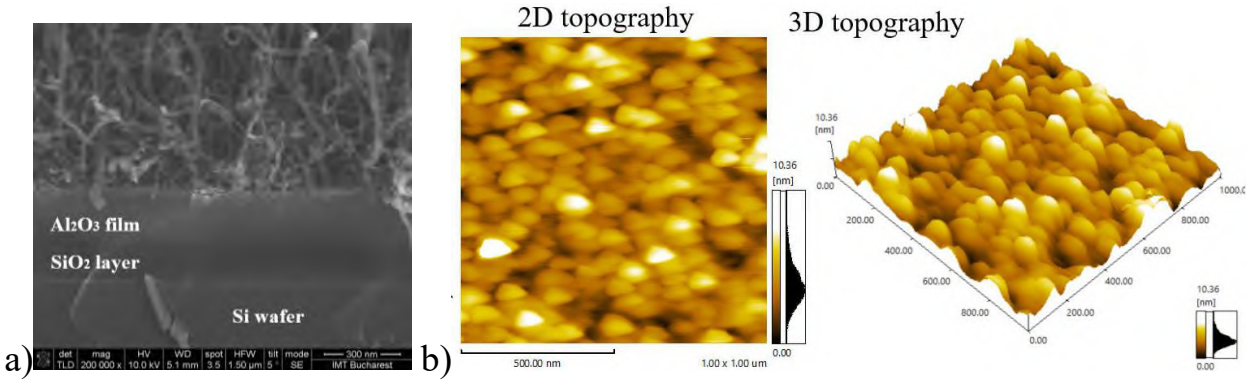
b) 

13-rasm. 750 C haroratda sintez qilingan UNNlari ko`ndalang (a) va sirt morfologiyasi (b).

Yuqori aniqlikdagi SEM tahlillari $Fe/Al_2O_3/SiO_2/Si$ geterostrukturasi yuzasida yuqori zichlikdagi, o‘ta kichik diametrli nanonaychalar massivi shakllanganini ko‘rsatdi. Olingan mikrofotografiyalarda nanonaychalar diametri bevosita o‘lchanganda, 3.6 va 4.1 nm o‘lchamdagi strukturalar aniqlanib, ular bir devorli uglerod nanonaychalariga (BDUNN), diametri 5.1 nm bo‘lganlari esa ikki devorli nanonaychalarga mos kelishi qayd etildi. Mazkur morfologik ko‘rsatkichlar Raman spektrlarida bir devorli nanonaychalar uchun xos bo‘lgan radial tebranish modalarining (RBM) mavjudligi hamda rentgen difraksiyasi (XRD) tahlillari orqali tasdiqlangan yuqori grafitlashgan kristallik darajasi bilan to‘liq o‘zaro moslikni namoyon etdi.

$Fe/Al_2O_3/SiO_2/Si$ geterostrukturasida sintez qilingan uglerod nanonaychalarining strukturaviy o‘lchamlari Raman spektroskopiyasi va yuqori aniqlikdagi SEM tahlillari orqali kompleks o‘rganildi. Raman spektrlarining past chastotali sohasida qayd etilgan radial tebranish modalaridan (RBM: 174, 227 va 268 cm^{-1}) nazariy hisoblangan diametrlar (0.93–1.43 nm) namunada bir devorli uglerod nanonaychalari (BDUNN) mavjudligini ko‘rsatdi. Yuqori

kattalashtirishdagi SEM tasvirlarida bevosita o‘lchangan nanonaychalar diametri ham (3.6 va 4.1 nm li BDUNN hamda 5.1 nm li IDUNN) ushbu natijalar bilan yaxshi mos kelib, sirtda o‘ta ingichka, kichik diametrli nanonaychalar shakllanganini eksperimental jihatdan to‘liq tasdiqladi.



a) b)

14-rasm. Fe/Al_2O_3/SiO_2/Si struktura sirtida sintez qilingan UNNlarning ko`ndalang kesim SEM tasviri (a) va taglikdan ajratib olingandan keyingi Al_2O_3 qatlamining AFM topografiyasi (b).

Aynan kichik diametrli BDUNNlarning selektiv o‘sishi Al_2O_3 oraliq bufer qatlamining sirt morfologiyasi bilan izohlandi. Taglik yuzasining AFM topografik tahlili termik ishlovdan so‘ng Al_2O_3 sirtida o‘rtacha notekisligi 1.061 nm, chuqurligi 10.353 nm gacha va kengligi 3–8 nm bo‘lgan nanoo‘lchamli relyef (nanoyamalar) hosil bo‘lishini ko‘rsatdi. Ushbu nano-chuqurliklar yuqori haroratda erigan Fe katalizatorining sirt bo‘ylab erkin diffuziyasini fazoviy cheklab, ularning o‘zaro qo‘shilib ketishi (koalesensiya va termik aglomeratsiya)ning oldini oladi. Natijada Fe nanozarrachalari o‘ta kichik va barqaror o‘lchamlarda saqlanib qolib, ular faqat kichik diametrli bir devorli uglerod nanonaychalarining nukleatsiyalanishi va o‘sishi uchun qulay katalitik sharoit yaratadi.

XULOSA

1. Elektron-nurli PVD usulida emissiya toki va bug‘latish vaqtini boshqarish orqali qalinligi 2,4–28,5 nm bo‘lgan Fe nanoqatlamlarini yuqori aniqlikda hosil qilish rejimlari aniqlandi. Qatlamlarning asosiy qismi kubik α-Fe metall fazasidan, sirti esa passivlashgan FeO va α-Fe_2O_3 oksid fazalaridan iborat ekanligi tasdiqlandi.

2. Termik ishlovda uzluksiz Fe qatlamining diskret nanozarrachalarga transformatsiyalanishi sirt diffuziyasi hodisasi bo‘yicha kechib, 8 nm qalinlikdagi Fe qatlami uchun to‘liq ajralishning chegara harorati 550 °C ekanligi aniqlandi. 750 °C haroratda barcha bufer qatlamlarda (SiO_2, Al_2O_3/SiO_2, TiO_2/SiO_2) diskret katalitik markazlar hosil bo‘lishining kritik qalinlik tuynugi 3–12 nm oralig‘ida bo‘lib, 20 nm va undan qalin qatlamlarda to‘liq parchalanish cheklanishi isbotlandi.

3. Al_2O_3(120 nm)/SiO_2(160 nm)/Si tizimida (700 °C, 5 daqiqa) shakllanadigan nanozarrachalar o‘rtacha diametri (d) va dastlabki Fe qatlami qalinligi (h) orasida $d \approx k \cdot h$ chiziqli bog‘lanish mavjud bo‘lib, empirik koeffitsiyent qiymati $k \approx 4–6$ oralig‘ida bo‘lishi belgilandi. Termik ishlov vaqtini 5 daqiqadan 15 daqiqagacha oshirish koalesensiya va Ostvald yetilishi jarayonlari tufayli nanozarralar o‘rtacha diametrining 32 nm dan 85 nm gacha ortishiga hamda sirt zichligining 83 dan 23 zarra/μm^2 gacha kamayishiga olib kelishi aniqlandi.

4. Fe nanozarrachalarining morfologik xususiyatlari taglik va bufer qatlamning strukturaviy tabiatiga bog‘liq ekanligi aniqlandi. Amorf SiO_2 va Al_2O_3/SiO_2 sirtlarida kichik va yuqori zichlikdagi kvazi-sferik zarralar shakllanib, ular anizotrop kristall sirtlaridagi qirralangan zarralarga nisbatan mos ravishda 2,4 va 2,0 marta kichik hamda 14 va 3,4 marta zichroq ekanligi ko`rsatildi.

5. UNN nukleatsiyasi jarayonida uglevodorod gazining nazoratsiz parchalanishi va konsentratsiyasi ortishi natijasida C atomlari katalizator zarrasi ichiga singib, Fe_3C fazasini hosil qilishi hamda faol markazlarni bloklab, katalizator faolligining yo‘qolishiga olib kelishi aniqlandi.

6. UNN devoridagi defektlarning katalizator bilan kuchli o‘zaro ta’siri natijasida katalizator aktiv qismi kamayishi aniqlandi va uning UNN terminatsiyasidagi ta’siri ochib berildi.

7. Fe (8–9 nm)/Al_2O_3 (120 nm)/SiO_2 (160 nm)/Si strukturada sintez qilingan UNNlarning nuqsonsizlik darajasi TiO_2/SiO_2 tizimiga nisbatan 19,2 %, tabiiy SiO_2 qatlamiga nisbatan 33,3 % va sapfir taglikka nisbatan 41,7 % yuqori bo‘lishi hamda past (550–650 °C) va yuqori (800–850 °C) harorat rejimlariga nisbatan 2,4–2,7 martagacha sifatliroq ekanligi isbotlandi.

8. Al_2O_3 sirtida termik ishlovda shakllanadigan nanochuqurchalar (R_a=1,06 nm) nisbatan qalin (8–9 nm) Fe qatlamlarida ham zarralar migratsiyasini cheklashi, diametri 0,93–1,43 nm bo‘lgan bir devorli UNNlar sintez qilish imkonini berishi aniqlandi. Ishlab chiqilgan Fe (8–9 nm)/Al_2O_3 (120 nm)/SiO_2 (160 nm)/Si katalitik tizimi yuqori strukturaviy sifatli bir va ko‘p devorli UNNlar olish hamda mustahkam kompozit izolyatorlar, sensorlar va nanoelektronika qurilmalarini yaratish uchun istiqbolli texnologik asos sifatida tavsiya etiladi.

SCIENTIFIC COUNCIL ON AWARDING OF SCIENTIFIC DEGREES DSc.05/25.27.12.FM/T.09.01 INSTITUTE OF ION-PLASMA AND LASER TECHNOLOGIES

INSTITUTE OF ION-PLASMA AND LASER TECHNOLOGIES

ISMATOV ASKAR ANVAR UGLI

PROCESSES OF NANOPARTICLE FORMATION AT THE CATALYST–BUFFER LAYER INTERFACE AND THEIR EFFECT ON CARBON NANOTUBE GROWTH

01.04.12 – Physics and Technologies of Nanomaterials

ABSTRACT OF DISSERTATION OF THE DOCTOR OF PHILOSOPHY (PHD) ON PHYSICAL AND MATHEMATICAL SCIENCES

Tashkent – 2026

The theme of the dissertation for doctor of philosophy (PhD) on physical and mathematical sciences was registered at the Supreme Attestation Commission under the Academy of Sciences of the Republic of Uzbekistan under number B2026.1.PhD/FM1481.

The doctoral (PhD) dissertation was carried out at the Institute of Ion-Plasma and Laser Technologiesof the Academy of Sciences of the Republic of Uzbekistan.

The abstract of the dissertation in three languages (Uzbek, Russian, English (resume)) has been posted on the website of the Scientific Council (www.iplt.uz) and on Information-educational portal "Ziyonet" information and educational portal at (http://www.ziyonet.uz).

Scientific supervisor:	**Ashurov Khatam Bakhronovich** Doctor of Technical Sciences, Professor
Official opponents:	**Tadjibayev Ilhom Ibrakhimovich** Doctor of Physical and Mathematical Sciences, Senior Researcher
	Hakberdiyev Elshod Olmosovich Doctor of Philosophy (PhD) in Physical and Mathematical Sciences, Senior Researcher
Leading organization:	National University of Uzbekistan named after Mirzo Ulugbek

The defense will take place on **«03» December 2026 at 16^{00}** at the meeting of the Scientific Council number DSc.05/2025.27.12.FM/T.09.01 at the Institute of Ion-Plasma and Laser Technologies (Address: 100125, Uzbekistan, Tashkent city, 33, Durmon yuli str., Phone: (99871) 262-32-54, e-mail: info@iplt.uz.).

The doctoral (PhD) dissertation can be looked through in the Information Resource Centre of the Institute of Ion-Plasma and Laser Technologies (is registered №26/18) (Address: 100125, 33, Durmon yuli str., Tashkent, Uzbekistan. Phone: (99871) 262-31-69.)

The abstract of dissertation was distributed on **« 20 » november 2026**.

(Mailing report № 26/18 on «20» november 2026).

Kh.B. Ashurov
Chairman of scientific councilon award of scientific degrees, Doctor of Technical Science, professor

I.D. Yadgarov
Scientific secretary of scientific council on award of scientific degrees, Doctor of Physical and Mathematical Sciences, professor

U.K. Makhmanov
Chairman of scientific seminar under scientific council on award of scientific degrees, Doctor of physical and mathematical sciences, professor

INTRODUCTION (PhD Dissertation Abstract)

Relevance and significance of the dissertation topic. Nowadays, modern materials science and nanotechnology place significant emphasis on the synthesis of nanoscale materials with controllable physical properties and their integration into practical functional devices. Among the most promising nanomaterials, carbon nanotubes (CNTs) possess superior mechanical strength, high electrical and thermal conductivity, chemical stability, and a large specific surface area, rendering them highly attractive for wide applications in sensors, energy storage devices, conductive composites, and other

functional systems. However, to ensure these practical characteristics of CNTs, precise control over their diameter, areal density, growth alignment, and structural quality is critically required.

In the chemical vapor deposition (CVD) method, the nucleation and growth of CNTs directly depend on the size, density, and stability of the catalyst nanoparticles. Although the Fe catalyst is highly effective in CNT synthesis due to its outstanding ability to decompose hydrocarbons and dissolve carbon, elevated synthesis temperatures often lead to nanoparticle coarsening, coalescence, substrate-directed diffusion, and carbon encapsulation (poisoning), resulting in a loss of catalytic activity. Therefore, determining the mechanisms and regularities governing the thermal dewetting of continuous Fe thin films into discrete nanoparticles represents an essential scientific problem in achieving controlled CNT synthesis.

The formation of Fe nanoparticles is governed not only by the initial film thickness and thermal annealing parameters, but also by the physicochemical nature of the supporting surface. Buffer layers such as SiO_2, Al_2O_3, and TiO_2 restrict the surface diffusion of Fe atoms, fundamentally dictating the size, morphology, and thermal stability of the resulting nanoparticles. Here, the thickness, structural state of the buffer layer, and its interfacial interaction with Fe serve as decisive factors. Nonetheless, the combined effects of Fe film thickness, thermal processing regimes, and buffer layer characteristics, as well as their quantitative correlation with CNT morphology and structural quality, remain insufficiently explored. From this perspective, investigating the transformation of Fe thin films deposited via electron-beam physical vapor deposition (EB-PVD) into nanoparticles on SiO_2, Al_2O_3, TiO_2, and sapphire substrates, establishing the regularities for controlling their size and areal density, and evaluating their subsequent influence on CNT nucleation and growth constitute a highly relevant scientific task. In this dissertation, the Fe film thickness, thermal annealing conditions, and buffer layer nature are comprehensively investigated as interrelated governing factors determining nanoparticle morphology.

In Uzbekistan, special attention is paid to the development of science, innovative technologies, and advanced materials. Introducing cutting-edge technologies into the economy, integrating scientific developments with industrial practice, and advancing materials science research represent key priorities. This dissertation research contributes directly to the implementation of tasks outlined in the Decree of the President of the Republic of Uzbekistan No. PF-165 dated July 6,

2022[4], Resolution No. PQ-390 dated December 27, 2025[5], Decree No. PF-21 dated February 16, 2026[6], as well as other relevant regulatory legal acts in the field.

Compliance of the research with priority areas of science and technology development of the Republic. This dissertation research was carried out in accordance with the priority direction of science and technology development in the Republic of Uzbekistan: "Physics, astronomy, energy, and mechanical engineering".

State of the art of the problem.

Controlling CNT parameters and the overall efficiency of the CVD process based on catalyst nanoparticle–substrate (buffer layer) interfacial interactions has been extensively investigated by both international and national scientific communities. In classical pioneer studies at the beginning of the 21st century (Z.P. Huang, J. Robertson), the formation of nanoparticles from thin films of transition metals (Ni, Co, Fe) upon thermal annealing and the potential to enhance their areal density on substrates such as TiN and SiO_2 were demonstrated. Subsequently, Suwen Li (2017) and other researchers showed that employing multilayer catalyst architectures enables effective regulation over nanoparticle size distributions.

Recent international research has predominantly focused on identifying the critical role of supporting buffer layers (Al_2O_3, TiO_2) during the thermal dewetting of metallic catalyst layers. It has been established that the Al_2O_3 buffer layer suppresses the surface diffusion and subsurface dissolution of metal atoms, significantly retarding nanoparticle coarsening induced by Ostwald ripening at elevated temperatures. Various research teams using EB-PVD, atomic layer deposition (ALD), and magnetron sputtering have confirmed that the tandem combination of an Fe catalyst with Al_2O_3/SiO_2 buffer layers provides precise control over the diameter and packing density of single-walled carbon nanotubes (SWCNTs) and vertically aligned multi-walled carbon nanotubes (VA-MWCNTs). Nevertheless, quantitative regularities concerning the phase evolution and interface-driven transformation of ultra-thin (sub-nanometer and nanometer-scale) Fe films into nanoparticles as a function of temperature and dwell time have not yet been fully resolved.

In Uzbekistan, an established scientific school has evolved regarding the synthesis and fundamental investigation of CNTs and nanostructures. At Qarshi State Technical University, under the leadership of Prof. A.A. Rakhmonkulov, experimental investigations on CNT synthesis via acetylene pyrolysis were initiated, while systemic research in this field has been established at the Arifov Institute of Ion-Plasma and Laser Technologies (IIPLT) of the Academy of Sciences of Uzbekistan. In particular, U. Khalilov and his research group have successfully conducted computer simulations of CNT nucleation and growth kinetics via molecular dynamics. Simultaneously, the Laboratory of Applied Nanotechnology at the Institute is conducting comprehensive applied and innovative research on the PVD/CVD fabrication of nanocatalysts and thin-film heterostructures,

[4] Decree of the President of the Republic of Uzbekistan, No. PF-165 dated October 22, 2024
[5] Resolution of the President of the Republic of Uzbekistan, No. PQ-390 dated December 27, 2025
[6] Decree of the President of the Republic of Uzbekistan, No. PF-21 dated February 16, 2026

morphological characterization, and their implementation in alternative energy technologies (lithium-ion battery anodes). However, further in-depth studies are required to comprehensively elucidate the atomistic formation mechanisms of EB-PVD-deposited Fe thin films on SiO_2, Al_2O_3, TiO_2, and sapphire substrates, as well as the interfacial phenomena governing the structural quality of CNTs.

Connection of the dissertation research with the scientific projects of the research institute. The research was carried out within the framework of fundamental project No. IL-5421101842: *"Nanocatalysts: formation, structure, size effects, and their influence on the synthesis of carbon nanomaterials"* at the Institute of Ion-Plasma and Laser Technologies.

Aim of the research. To establish the formation mechanisms of physical vapor-deposited Fe nanoparticles on various oxide structures and substrates, and to evaluate the influence of the physicochemical nature and parameters of buffer layers on carbon nanotube growth under diverse conditions.

Research objectives:

to develop operational regimes for controlling the thickness of Fe thin films deposited on SiO_2, Al_2O_3, and TiO_2 buffer layers via electron-beam PVD by adjusting evaporation time and emission current, and to determine the elemental and phase composition of the films;

to establish the stages and boundary conditions governing the transition of a continuous Fe thin film into a system of discrete nanoparticles upon thermal annealing;

to quantitatively evaluate the influence of Fe film thickness, annealing temperature, and dwell time on nanoparticle size, areal density, and size distribution on SiO_2, Al_2O_3, and TiO_2 nanostructures, and to derive an empirical relationship between nanoparticle diameter and initial film thickness;

to investigate the correlation between CNT nucleation/growth kinetics and catalyst nanoparticle characteristics, including growth modes and the physicochemical origins of catalyst deactivation;

to perform a comparative analysis of the structural quality and morphology of CNTs synthesized across various catalytic systems ($Fe/SiO_2/Si$, $Fe/Al_2O_3/SiO_2/Si$, $Fe/TiO_2/SiO_2/Si$, and Fe/sapphire), determining the optimal catalyst–buffer layer architecture;

to investigate the catalytic deactivation mechanisms of Fe nanoparticles caused by high-temperature interfacial interactions with buffer layers and to identify the optimal temperature window for CNT synthesis.

Object of the Research. Fe nanoparticles synthesized on SiO_2, Al_2O_3, and TiO_2 buffer layers and sapphire substrates, as well as the CNTs grown from these catalytic systems.

Subject of the Research. The formation processes of Fe nanoparticles on various oxide buffer layers, optimization of thermal annealing parameters controlling nanoparticle size and spatial distribution, the nucleation kinetics and deactivation mechanisms of CNTs on catalyst surfaces, and the synthesis processes of single-walled carbon nanotubes.

Research Methods. Dry thermal oxidation was employed to grow SiO_2 layers on silicon substrates; electron-beam physical vapor deposition (EB-PVD) was used for depositing Al_2O_3 and Fe thin films; atomic layer deposition (ALD) was applied for TiO_2 film fabrication; and chemical vapor deposition (CVD) was utilized for CNT synthesis. The structural and morphological properties of the obtained samples were systematically characterized using AFM, XPS, XRD, TEM, SEM, and Raman spectroscopy.

Scientific Novelty of the Research:

For the first time, it was demonstrated that high-density (≈ 28 nm, 302 particles/μm^2), quasi-spherical nanoparticles form on amorphous SiO_2 and Al_2O_3 surfaces, whereas faceted and comparatively sparse particles develop on crystalline sapphire and TiO_2 surfaces (≈ 68 nm, 17 particles/μm^2 and ≈ 56 nm, 87 particles/μm^2, respectively). Concurrently, it was proven that the non-uniform surface distribution and coarsening of the particles on these crystalline substrates stem from a weaker suppression of surface Fe atom diffusion compared to the amorphous Al_2O_3 layer;

In the Al_2O_3 (120 nm)/SiO_2 (160 nm)/Si system, the empirical relationship $d \approx k \cdot h$ was established between the average diameter (d) of nanoparticles formed upon thermal annealing of an electron-beam PVD-deposited Fe thin film at 700 °C for 5 minutes and the initial film thickness (h); **for the first time**, the broad scaling range reported in the literature for Fe-based systems ($k \approx 2$–10) was narrowed down under these conditions to $k \approx 4$–6 (with $k \approx 5$), enabling predictive estimation and control of catalyst nanoparticle dimensions via the initial film thickness for CNT growth";

For the first time, a comprehensive comparative analysis of CNTs synthesized on SiO_2, Al_2O_3/SiO_2, and TiO_2/SiO_2 buffer layers as well as sapphire substrates was performed. The structural quality of CNTs synthesized on the Al_2O_3 (120 nm)/ SiO_2 (160 nm) tandem proved superior to those grown on TiO_2 by 19.2%, on native SiO_2 by 33.3%, and on sapphire by 41.7%;

For the first time, the synthesis of single-walled carbon nanotubes (SWCNTs, $d = 0.9$–1.1 nm) was successfully achieved even from relatively thick (8–9 nm) Fe catalyst layers on the Al_2O_3(120 nm)/SiO_2(160 nm) structure. It was revealed that the nanoscale surface roughness ($R_a = 1.06$ nm) of the Al_2O_3 buffer layer, coupled with its relatively strong metal–support interaction with Fe particles, renders it the most effective buffer layer at an annealing temperature of 700 °C.

Practical results of the research. The findings provide optimal physical and chemical processing regimes for producing Fe nanoparticles with an average diameter of 25–30 nm and an areal density of ≈ 300 particles/μm^2 on various oxide buffer layers. Furthermore, methods for precisely controlling nanoparticle dimensions by adjusting the thickness of catalytic and buffer layers, annealing temperature, and duration have been established. These nanoparticles can be actively utilized as high-efficiency catalysts in methane and hydrogen cracking, ammonia synthesis, carbon nanofiber and boron nitride nanotube synthesis, and photoelectrochemical water splitting. Additionally, experimental conditions for

synthesizing vertically aligned, single-walled, and multi-walled CNTs catalyzed by Fe nanoparticles were validated, and design parameters for advanced catalyst architectures were formulated to maximize CNT structural quality and array height.

Reliability of the results. The reliability of the results is ensured by the application of standardized modern synthesis methods, high-precision analytical instrumentation, a statistically sufficient volume of experimental data, and the rigorous reproducibility of experimental findings.

Scientific significance of the research results. The scientific significance lies in uncovering the regularities governing the morphological transformation of EB-PVD Fe thin films into nanoparticles on SiO_2, Al_2O_3, TiO_2, and sapphire substrates, and quantitatively determining the controlling factors (film thickness, annealing temperature, duration, and substrate nature), which establish the scientific foundation for size-controlled nanocatalyst fabrication. Furthermore, the interfacial mechanisms dictating CNT nucleation, growth kinetics, and structural quality were elucidated, and the Al_2O_3/SiO_2 tandem buffer layer was experimentally validated as a superior barrier preserving catalytic activity under high-temperature conditions (850 °C).

Practical significance of the research results. The practical significance is defined by establishing precision deposition regimes (evaporation duration and emission current) for Fe catalyst films and determining the optimal thermal annealing conditions to fabricate high-density, uniform Fe nanoparticles. The feasibility of synthesizing high-structural-quality ($I_D/I_G = 0.42$), single-walled CNTs (diameters of 0.93–1.43 nm) on the Fe (8–9 nm)/Al_2O_3 (120 nm)/ SiO_2 (160 nm)/Si heterostructure was experimentally demonstrated. The obtained results serve as practical guidelines for engineering catalyst–buffer layer systems tailored for CNT-based energy storage systems, conductive composites, and sensory devices.

Implementation of the research results.

The scientific developments and conclusions obtained within the framework of the dissertation titled *"Processes of nanoparticle formation at the catalyst–buffer layer interface and their effect on carbon nanotube growth"* have been implemented in the following areas:

The findings were applied to fundamental project AL-7623031231: *"Synthesis of carbon nanotubes based on cyclopentadienyliron dicarbonyl dimer and investigation of their physicochemical properties"*, carried out in 2024–2025 at the Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan within the framework of Uzbekistan–Turkmenistan scientific cooperation (Reference № 4/1255-3131 from the Academy of Sciences of the Republic of Uzbekistan dated November 4, 2026). The utilization of optimal kinetic conditions for generating high-density, uniformly distributed Fe nanoparticles on Al_2O_3, TiO_2, and SiO_2 buffer layers enabled the controlled synthesis of highly crystalline, high-quality single-walled carbon nanotubes with precisely tuned catalytic active sites;

The established regularities concerning the CVD synthesis of CNTs on $Fe/Al_2O_3/SiO_2/Si$, $Fe/TiO_2/SiO_2/Si$, and $Fe/SiO_2/Si$ heterostructures, as well as the effects of buffer layer nature, thermal regimes, and dwell times on resulting nanostructure morphology and quality, were utilized in the execution of the applied project *"Improving the efficiency of silicone-polymer-based IPP-10/1000-8A UHL1 and LK 70/10-3 UXL1 high-voltage insulators"* (2024–2026) at the Joint Venture LLC "Armatura-Izolyator Zavodi (AIZ)" (Reference No. 19 from JV LLC "AIZ" dated January 16, 2026). Integrating these findings ensured optimal loading and uniform dispersion of carbon nanotubes within the silicone-polymer matrix, significantly enhancing the operational endurance, mechanical durability, and resistance of the electrical insulators against aggressive environmental factors.

Approbation of the research results. The primary results of the research were presented and discussed at 3 national and 4 international scientific conferences.

Publication of the research results. A total of 12 scientific works have been published on the main outcomes of the dissertation, including 2 papers in international peer-reviewed journals and 3 articles in national scientific journals recommended by the Supreme Attestation Commission (OAK) for publishing the main scientific results of doctoral dissertations.

Structure and Volume of the Dissertation.

The dissertation consists of an introduction, 4 chapters, conclusions, a list of references, and appendices. The main text comprises 117 pages, containing 56 figures and 7 tables.

MAIN CONTENT OF THE DISSERTATION

The Introduction substantiates the relevance of the topic regarding the formation of Fe nanoparticles at the catalyst–buffer layer interface and their effect on carbon nanotube growth, outlining the aim, objectives, object, subject, and applied research methods of the study. It highlights the compliance of the work with the priority directions of science and technology development in the Republic. Furthermore, comprehensive information is provided on the scientific novelty, theoretical and practical significance, reliability, approbation, and publications of the obtained results, as well as the overall structure and scope of the dissertation**.**

The first chapter of the dissertation, entitled "Literature review and current state of the scientific problem," provides a comprehensive analysis of scientific literature concerning the structure, properties, and growth mechanisms of carbon-based nanomaterials, specifically focusing on 0D, 1D, 2D, and 3D carbon nanostructures, their chirality types, and prospective areas of practical application. The chapter presents a comparative review of the primary CNT synthesis techniques—arc discharge, laser ablation, and CVD methods—delineating their respective advantages and limitations. The crucial role of Fe, Ni, and Co nanocatalysts in CNT growth is systematically detailed, particularly emphasizing the factors influencing the morphology and catalytic activity of Fe-based catalysts, including nanoparticle size, substrate nature, and thermal annealing parameters. Methods for fabricating Fe nanostructures (PVD, ALD, magnetron sputtering) are

compared, evaluating their distinct technological capabilities. In the concluding section of the chapter, literature data correlating the CVD method with technological growth parameters (temperature, dwell time, and catalyst thickness) are generalized, explicitly substantiating the gaps in existing research—specifically, the lack of comprehensive quantitative understanding regarding the atomic-scale formation regularities of electron-beam PVD-deposited Fe thin films on diverse oxide buffer layers.

The second chapter, entitled "Optimal conditions for fabricating buffer–catalyst layers and experimental and analytical methods for carbon nanotube synthesis," describes the starting materials utilized in the study—single-crystal silicon (KES, (100) orientation, 375 μm thickness) and sapphire substrates—along with their preparation procedures. The chapter elaborates on the growth of SiO_2 layers via dry thermal oxidation and the measurement of their thickness using spectroscopic ellipsometry, the deposition of Al_2O_3 layers on Si/SiO_2 structures via electron-beam PVD, the synthesis of TiO_2 buffer layers via atomic layer deposition (ALD), and the electron-beam evaporation of Fe catalytic thin films. In addition, the CVD synthesis technology and optimal operational parameters for carbon nanotubes are presented, followed by the operating principles and measurement parameters of the analytical characterization techniques employed to evaluate structural and morphological properties, including SEM, TEM, STEM, AFM, XRD, XPS, FTIR, and Raman spectroscopy.

The third chapter, entitled **"Formation of Fe nanostructures and analysis of influencing factors,"** investigates the phase composition and structural state of physical vapor-deposited Fe thin films formed on Al_2O_3 and SiO_2 buffer layers using SEM, EDX, XPS, and XRD techniques. The XPS spectra revealed that the Fe atoms exist predominantly in the metallic state, with the possibility of partial surface oxidation (Figure 1).

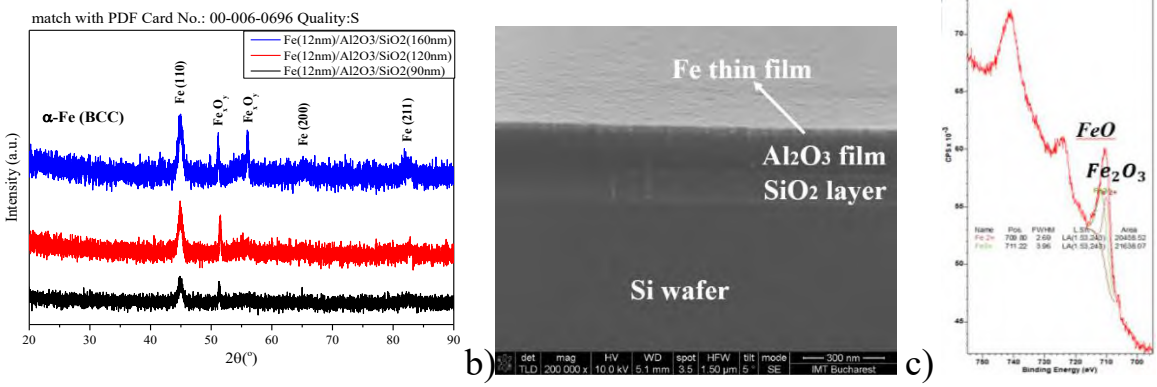


Figure 1. XRD analysis (a), cross-sectional SEM image (b), and XPS analysis (c) of the thin Fe film.

The mechanism governing the formation of nanostructures from the catalytic thin film upon thermal annealing was investigated; as the temperature increased, the continuous 8 nm-thick Fe film underwent thermal dewetting into a system of discrete nanoparticles. Atomic force microscopy (AFM) images demonstrated that at approximately 550 °C, the continuous film completely disintegrated, yielding near-

spherical nanoparticles (Figure 2). Furthermore, in-depth investigations into the transformation from a continuous layer to discrete nanoparticles revealed that thermal heating initially enhances atomic vibrations, triggering interfacial instability and localized void formation at energetically unfavorable sites. In the subsequent stage, pinholes nucleate on the surface, which expand to form rims, followed by the development of discrete nano-islands. Driven by surface energy minimization and surface diffusion, mass transport of atoms occurs, a kinetic process that was described following the Arrhenius law.

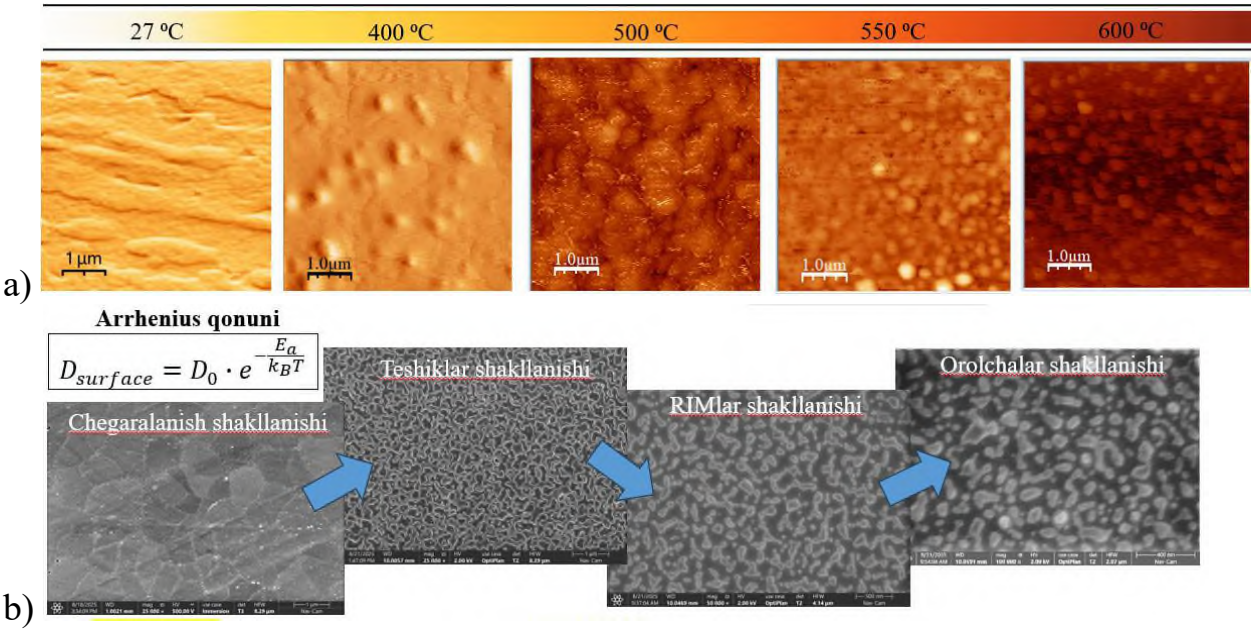


Figure 2. a) AFM and b) SEM images of the transformation process of an 8 nm-thick continuous Fe thin film into discrete nanoparticles.

SEM analyses illustrating the significance of film thickness in nanostructure formation, as well as the effects of thermal annealing temperature and dwell time on the morphology of the resulting nanoparticles, are presented:

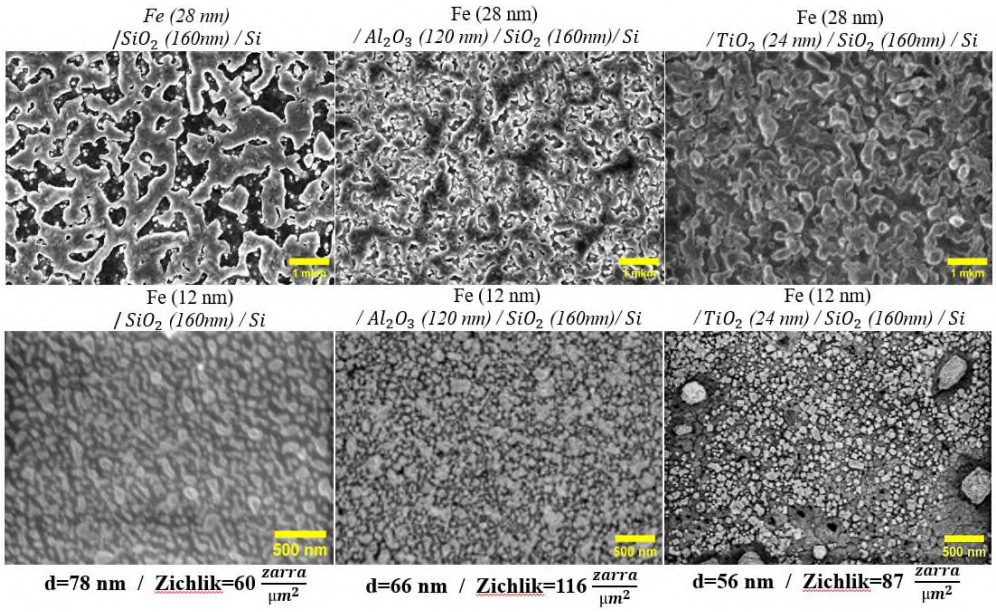


Figure 3. SEM images of the surface morphology of Fe thin films with various thicknesses on different substrates after thermal annealing at 700 °C for 10 minutes.

28 nm-thick Fe films subjected to thermal annealing at 700 °C for 10 minutes did not undergo complete dewetting, forming irregular morphologies, whereas 12 nm-thick films yielded a substantially smaller and homogeneous system of nanoparticles (Figure 3).

Here, the resulting average nanoparticle sizes were determined to be 78 nm on the SiO_2 surface, 66 nm on the Al_2O_3/SiO_2 surface, and 56 nm on the TiO_2/SiO_2 surface. In investigations aimed at controlling nanoparticle sizes, Fe thin films with thicknesses of 3–20 nm were deposited onto SiO_2/Si substrate surfaces via the EB-PVD technique. The nanoparticles formed upon thermal annealing of these thin films at 700 °C for 5 minutes were analyzed using SEM (Figure 4). It was established that an 8–9 nm-thick thin film yields nanoparticles with an average size of 25–30 nm and an areal density of 300 particles/μm^2.

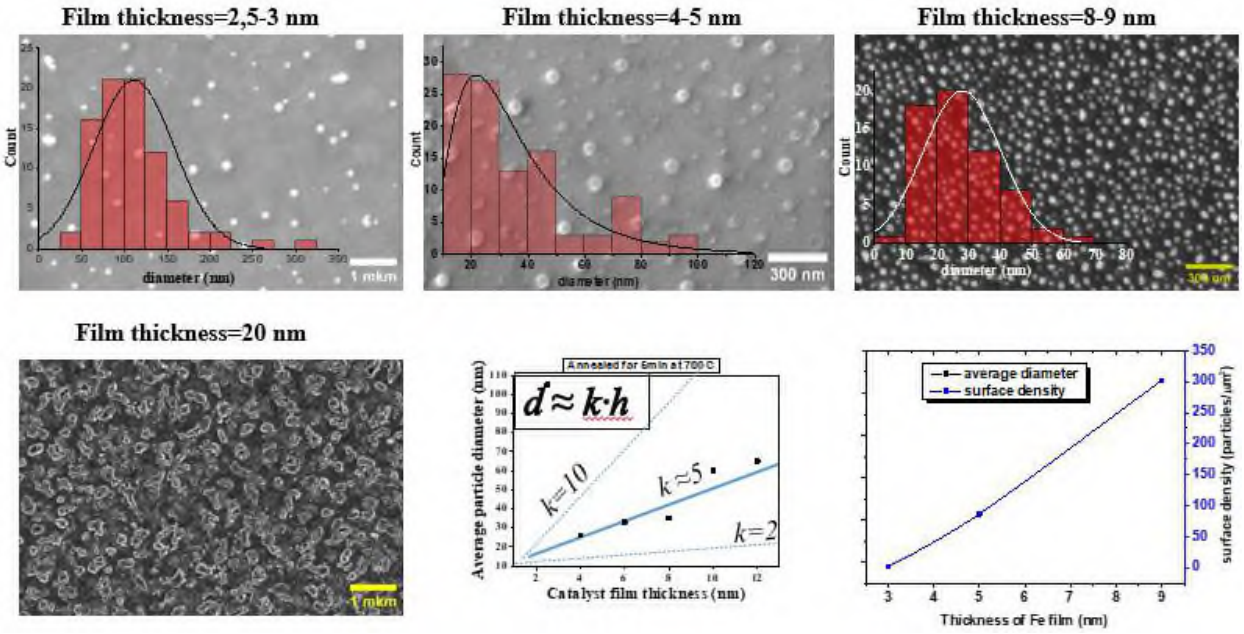


Figure 4. Morphology of nanoparticles formed on the SiO_2/Si surface from Fe thin films of various thicknesses after thermal annealing at 700 °C for 5 minutes.

Furthermore, the empirical relationship $d = k \cdot h$ correlating nanoparticle diameter (d) with initial thin film thickness (h) was evaluated, effectively narrowing down the scaling factor k from the widely reported literature range of [2–10] to a defined interval of [4–6]. This finding substantially enhances precision in synthesizing nanoparticles of predetermined dimensions, enabling direct and predictable control over nanoparticle size via the initial film thickness.

In investigations conducted across various temperatures to elucidate the effect of thermal annealing temperature on nanoparticle size control, the 700–750 °C temperature window was determined to provide the optimal conditions for forming Fe nanoparticles with the smallest diameters and the highest areal density. At temperatures of 800 °C and above, a pronounced increase in nanoparticle

dimensions was confirmed, driven by accelerated surface atom migration, Ostwald ripening, and coalescence processes.

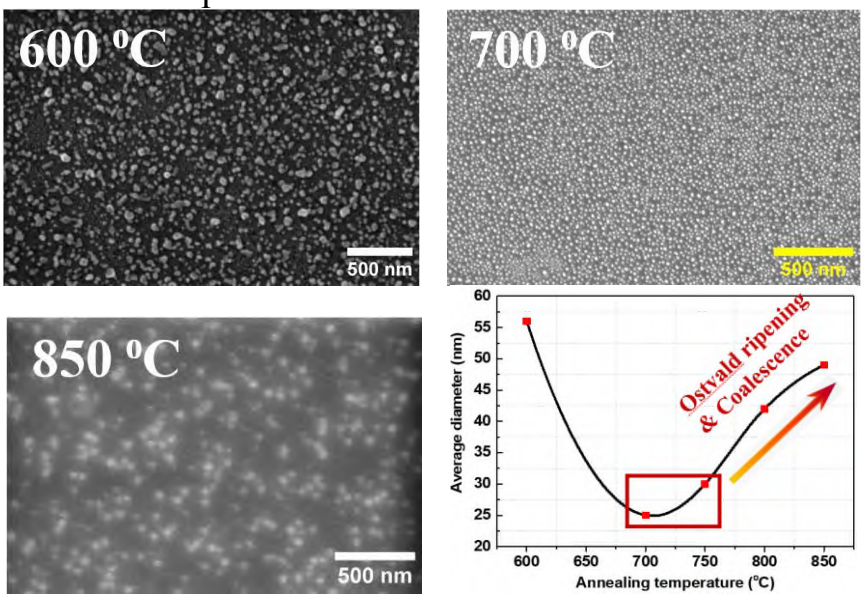
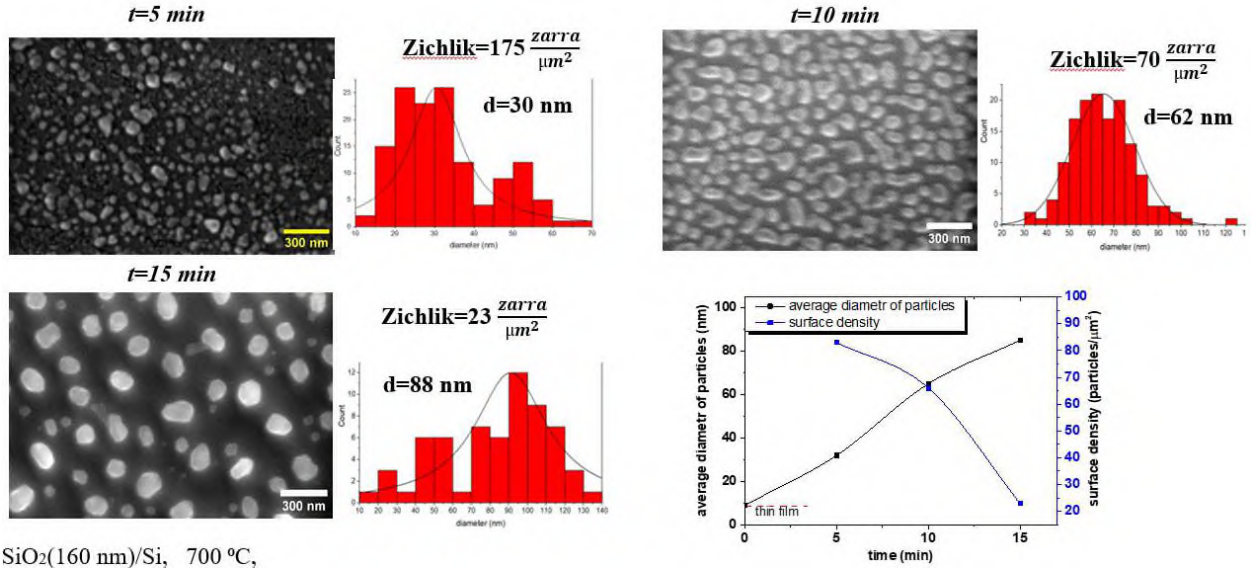


Figure 5. Surface morphology of nanoparticles formed from an 8–9 nm-thick Fe thin film annealed at various temperatures and their corresponding temperature dependence plot.

Nanoparticle size depends not only on the initial film thickness and annealing temperature, but also on the annealing duration. The findings led to the following conclusions: thermal annealing duration cannot be extended arbitrarily, as excessive dwell times significantly increase the probability of nanoparticles coalescing with neighboring atoms. Experimental results revealed that after 10 minutes of annealing, the majority of the particles underwent coalescence, resulting in a twofold increase in average diameter and a more than twofold reduction in areal density compared to a 5-minute treatment.



SiO_2(160 nm)/Si, 700 °C,

Figure 6. SEM images and surface size distribution histograms of nanoparticles formed from an 8–9 nm-thick Fe thin film annealed at 700 °C for various durations.

Prolonging the annealing duration to 15 minutes led to nearly a threefold increase in diameter and a sevenfold decrease in particle density relative to the 5-minute regime.

As established from the SEM micrographs, the nanoparticles formed on the surfaces of the aforementioned amorphous SiO_2 and Al_2O_3 layers exhibit spherical and quasi-spherical shapes.

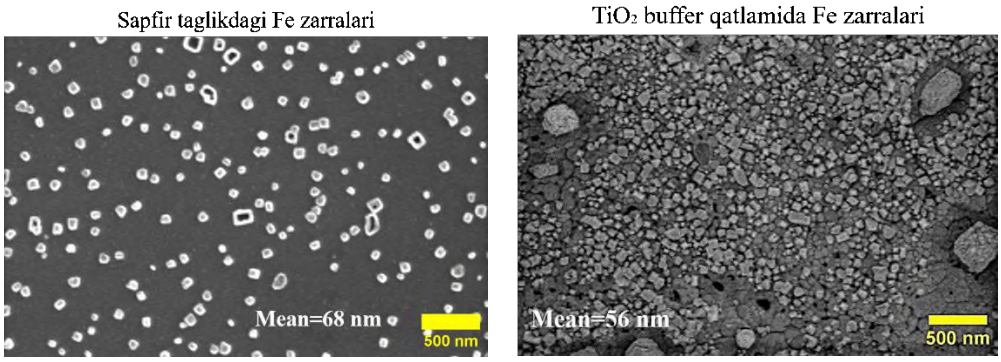


Figure 7. Fe nanoparticles formed on the surface of sapphire and TiO_2 buffer layers.

However, investigations regarding the formation of Fe nanoparticles on crystalline substrates remain scarce in the current literature. The morphology of nanoparticles formed upon thermal annealing of an 8–9 nm-thick Fe thin film on sapphire and anatase-phase TiO_2 buffer layers indicates that (Figure 7) the average particle size and areal density are 68 nm and 22 particles/μm^2 on the sapphire substrate, and 56 nm and 87 particles/μm^2 on the TiO_2 buffer layer, respectively. Compared to SiO_2 and Al_2O_3, these results were attributed to enhanced surface diffusion of atoms on the TiO_2 buffer layer and sapphire substrate, which served as the primary factor driving particle coarsening. Another notable aspect is the faceted geometry of the particles, which was explained by the crystalline template effect of the underlying substrate.

The fourth chapter, entitled "Effect of catalyst nanoparticle and buffer layer parameters and physicochemical nature on CNT synthesis," analyzes the correlation between CNT nucleation and growth mechanisms and catalyst nanoparticles: the adsorption of carbon atoms onto the catalyst surface, their bulk diffusion into the nanoparticle, and precipitation in the form of a graphitic phase upon reaching critical supersaturation were investigated.

Cross-sectional SEM images revealed catalyst nanoparticles positioned at the tips of the nanotubes, confirming that CNT growth proceeded via the tip-growth mechanism. SEM and TEM analyses of the sample synthesized via the CVD method (600 °C, 5 min, $Ar/C_2H_2 = 2:2$) demonstrated that the nucleation stage on catalyst nanoparticles represents the decisive phase in carbon nanotube (CNT) growth. Experimentally, thin CNTs grew from only a fraction of the particles, whereas the majority failed to evolve into active growth centers (Figure 8a).

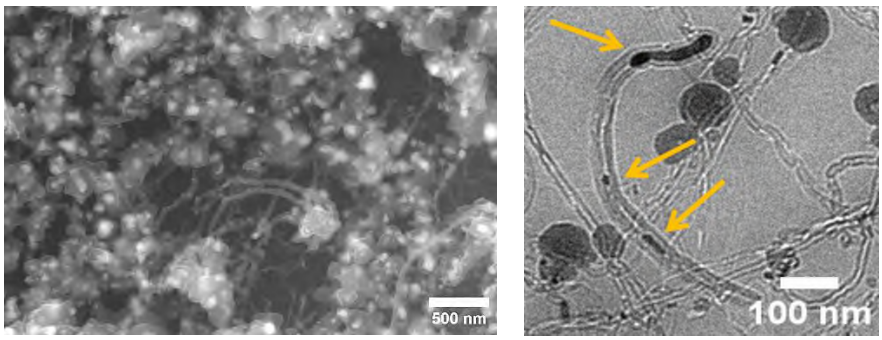


Figure 8. a) SEM image of CNTs synthesized at 600 °C under an $Ar/C_2H_2 = 2:2$ gas flow, and b) TEM image of CNTs synthesized at 700 °C under an $Ar/C_2H_2 = 7:2$ gas flow.

This behavior was attributed to a kinetic imbalance between the carbon supply rate at the catalyst surface and the bulk diffusion rate within the nanoparticle: instead of dissolving and diffusing into the nanoparticle bulk to reach supersaturation, carbon accumulated excessively on the outer surface, forming passivating "core–shell" or carbon nano-onion morphologies (catalyst encapsulation/poisoning). Furthermore, TEM analyses confirmed that structural defects in the growing CNT walls interact strongly with the catalyst nanoparticle, tearing off portions of the active catalytic mass and directly inducing growth termination.

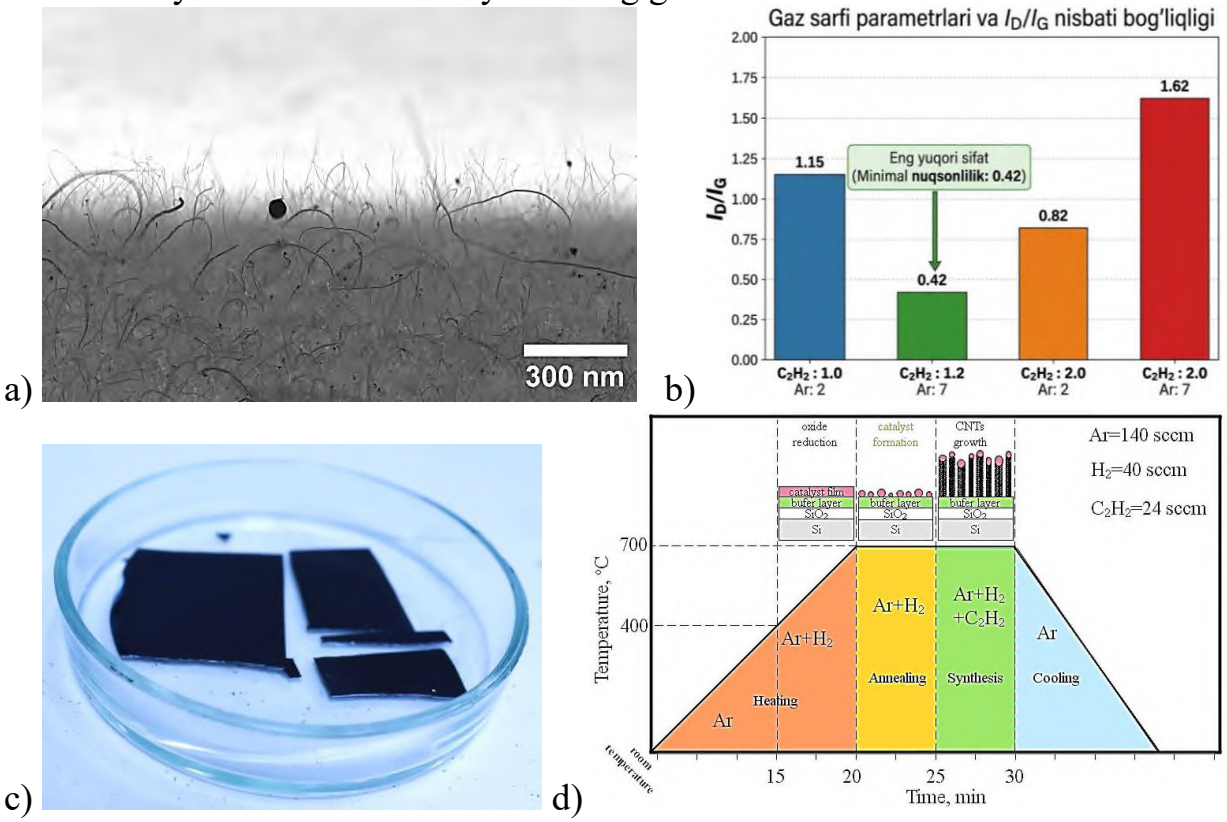


Figure 9. Cross-sectional SEM image of CNTs formed via the tip-growth mechanism (a), plot of CNT structural quality versus gas flow parameters (b), photograph of synthesized CNTs on a Si substrate (c), and timeline chronology of the CVD process (d).

These experimental morphological observations show complete agreement with atomistic simulation models describing carbon accumulation, supersaturation, and nucleation mechanisms, demonstrating the critical necessity of precisely

regulating the kinetic balance between surface decomposition reactions and bulk diffusion to achieve high-density, high-quality CNT growth.

Experimental results demonstrated that the growth of CNTs proceeded predominantly via the VLS (Vapor–Liquid–Solid) and tip-growth mechanisms. Cross-sectional SEM micrographs revealed catalyst nanoparticles (contrast points) located at the tips of the growing nanotubes, possessing dimensions comparable to or slightly exceeding the nanotube diameters. The detachment and upward lift of the nanoparticles with the growing nanotube tips—rather than remaining anchored to the substrate surface (*base-growth*)—indicates a relatively weak adhesive interaction between the catalyst and the underlying substrate; consequently, carbon diffusion within the quasi-liquid nanoparticle and subsequent supersaturation detached the nanoparticle from the substrate, driving the tip-growth regime.

Raman spectroscopy analyses revealed that the precursor gas component ratio (C_2H_2:Ar) exerted a significant influence on the structural perfection of the CNTs. Under the C_2H_2:Ar $= 1.2:7$ regime, the I_D/I_G ratio reached a minimum value of 0.42, corresponding to the highest degree of crystallinity and minimal defect density. Conversely, increasing the acetylene flow rate (C_2H_2:Ar $= 2.0:7$) drastically increased the defect density ($I_D/I_G = 1.62$) due to the excessive accumulation and deposition of amorphous carbon on the surface.

The effects of the substrate and buffer layer nature on catalyst morphology and the resulting structural quality of the carbon nanotubes (CNTs) were comparatively investigated using SEM and Raman spectroscopy.

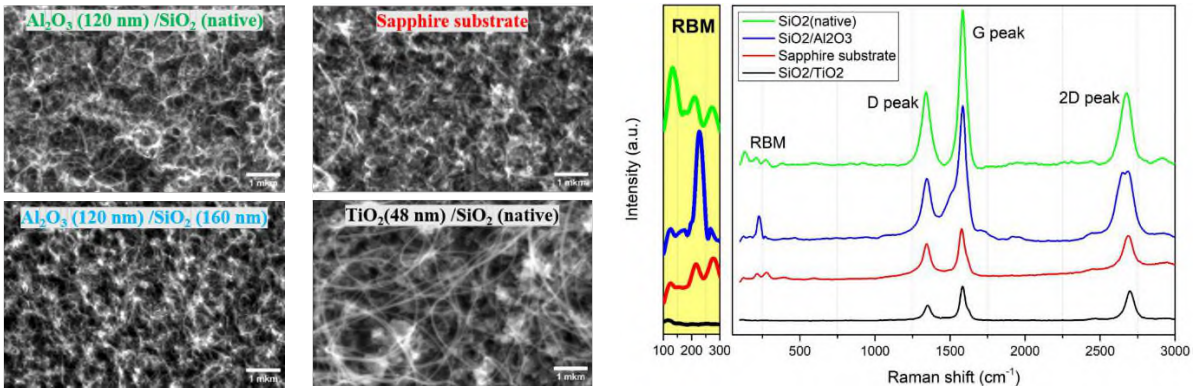


Figure 10. Surface morphology (a) and Raman spectra (b) of CNTs synthesized on various substrates.

Analyses revealed that the Fe (8–9 nm)/Al_2O_3 (120 nm)/SiO_2 (160 nm)/Si heterostructure serves as the most optimal catalytic system. In this architecture, the thick SiO_2 and Al_2O_3 buffer layers effectively suppressed the thermal agglomeration of catalyst nanoparticles, ensuring their high dispersion and uniform areal distribution; consequently, single-walled carbon nanotubes (diameters of 0.93–1.43 nm) with the highest array density and lowest defect level ($I_D/I_G = 0.42$) were obtained. For comparison, nanoparticles formed on the Fe (8–9 nm)/TiO_2 (48 nm)/ SiO_2 (4 nm)/Si structure exhibited a faceted morphology; since coalescence was not completely hindered, the resulting nanotubes possessed larger diameters and a higher defect density ($I_D/I_G = 0.52$). In the Fe (8–9 nm)/Al_2O_3 (120 nm)/ SiO_2 (4 nm)/Si structure with a native oxide layer, the structural quality degraded

($I_D/I_G = 0.63$, diameters of 0.91–1.25 nm) due to insufficient dispersion control. The highest defect density was observed on the Fe/sapphire substrate ($I_D/I_G = 0.72$, diameters of 0.90–1.20 nm), which was attributed to the formation of coarse and sparsely distributed Fe nanoparticles under the templating influence of the sapphire crystal lattice.

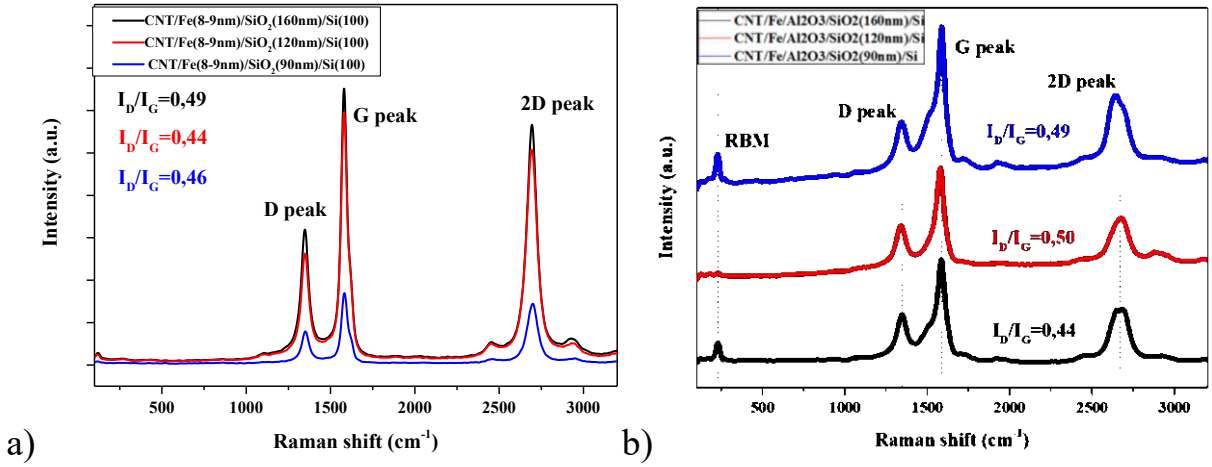


Figure 11. Raman spectra of CNTs synthesized at 700 °C on SiO_2 and Al_2O_3/SiO_2 structures of various thicknesses.

Thus, among the investigated catalytic systems, the $Fe/Al_2O_3/SiO_2/Si$ heterostructure was determined to be the most effective foundation for mitigating thermal agglomeration, efficiently controlling nanoparticle dimensions, and enabling the targeted synthesis of high-quality single-walled CNTs.

The influence of the SiO_2 dielectric layer thickness (90, 120, and 160 nm) on CNT structural quality was evaluated via Raman spectroscopy during the CVD process (700 °C, 5 min). In all samples, characteristic Raman bands of the sp^2-hybridized carbon lattice ($D \approx 1350$ cm^{-1}, $G \approx 1580$ cm^{-1}, and $2D \approx 2700$ cm^{-1}) were detected, with the defect ratio varying within a narrow interval ($I_D/I_G \approx 0.44$–0.49). The lowest defect density was recorded for the 120 nm-thick SiO_2 layer ($I_D/I_G \approx 0.44$), whereas a slightly higher value was observed for the 160 nm-thick layer ($I_D/I_G \approx 0.49$); however, it was established that variations in SiO_2 thickness do not exert a drastic or fundamental influence on the crystallinity of the nanotubes.

Incorporating the multilayer Fe (8–9 nm)/Al_2O_3 (120 nm)/SiO_2/Si heterostructure to suppress the thermal agglomeration of Fe nanoparticles and maintain their stable dispersion at elevated temperatures considerably improved the graphitization degree ($I_D/I_G < 0.5$). Most importantly, the emergence of radial breathing modes (RBM) in the low-frequency region (100–300 cm^{-1}) confirmed that the Al_2O_3 buffer layer effectively facilitates not only high graphitic ordering, but also the formation of single-walled carbon nanotubes (SWCNTs).

To evaluate the thermal stability of the catalytic system under high-temperature hydrocarbon pyrolysis conditions (e.g., CH_4, C_2H_4), comparative CNT synthesis was performed on $Fe/SiO_2/$Siand $Fe/Al_2O_3/SiO_2/Si$ structures at 850 °C.

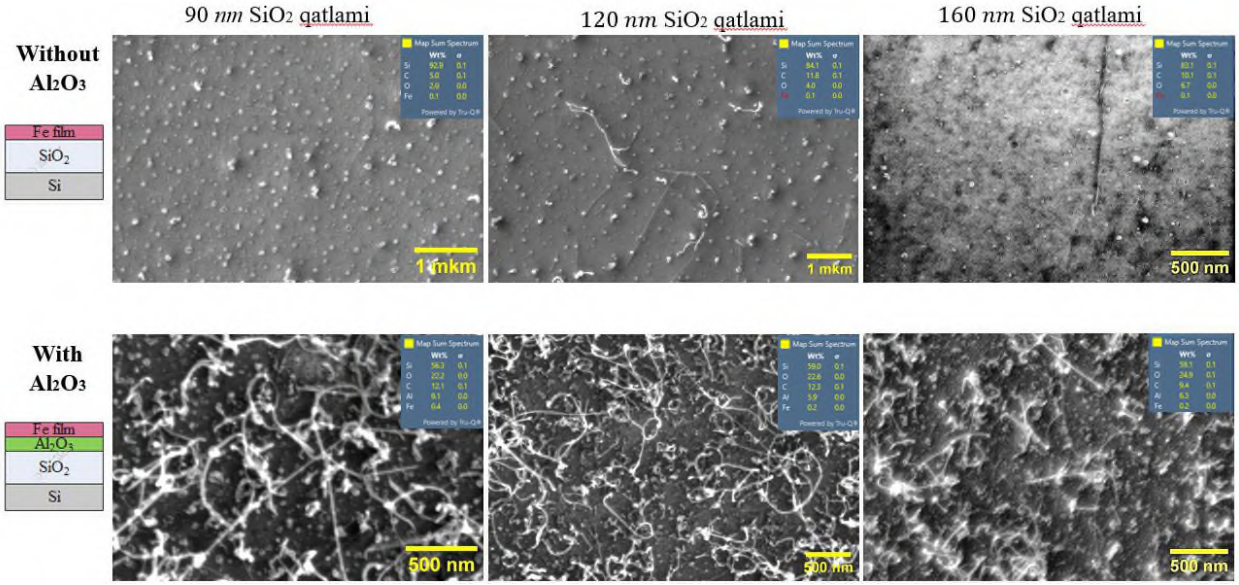


Figure 12. Surface morphology of CNTs synthesized at 850 °C on $SiO_2$and Al_2O_3/SiO_2structures of various thicknesses.

The analyses revealed that nanotube growth was virtually suppressed on samples containing only the SiO_2buffer layer; this was attributed to the high-temperature accelerated diffusion of Si atoms leading to the formation of catalytically inactive iron silicide phases (Fe_xSi_y, such as FeSiand $FeSi_2$), as well as severe particle coalescence and deactivation. In contrast, in the heterostructure incorporating an intermediate Al_2O_3buffer layer, both the particle size and active dispersion were fully maintained even at 850 °C, facilitating the growth of high-density, entangled CNT networks across the surface. This is directly associated with the ability of the Al_2O_3layer to act as an effective diffusion barrier against silicide formation while mitigating thermal agglomeration, confirming the superior structural and chemical stability of this multilayer system over a wide temperature range.

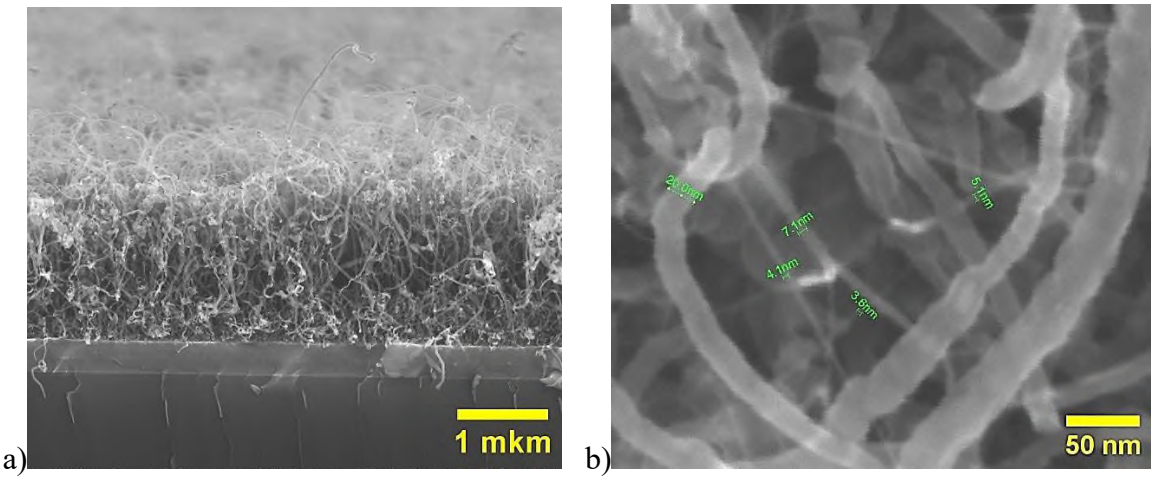


Figure 13. Cross-sectional (a) and surface morphology (b) of CNTs synthesized at 750 °C.

High-resolution SEM analyses demonstrated the formation of a high-density array of ultra-small-diameter nanotubes on the surface of the $Fe/Al_2O_3/SiO_2/Si$ heterostructure. Direct diameter measurements from the obtained micrographs

revealed structures with dimensions of 3.6 and 4.1 nm, corresponding to single-walled carbon nanotubes (SWCNTs), as well as nanotubes with a diameter of 5.1 nm, attributed to double-walled carbon nanotubes (DWCNTs). These morphological characteristics exhibited complete consistency with the radial breathing modes (RBM) characteristic of single-walled nanotubes in the Raman spectra, as well as the high degree of graphitic crystallinity confirmed by X-ray diffraction (XRD) analysis.

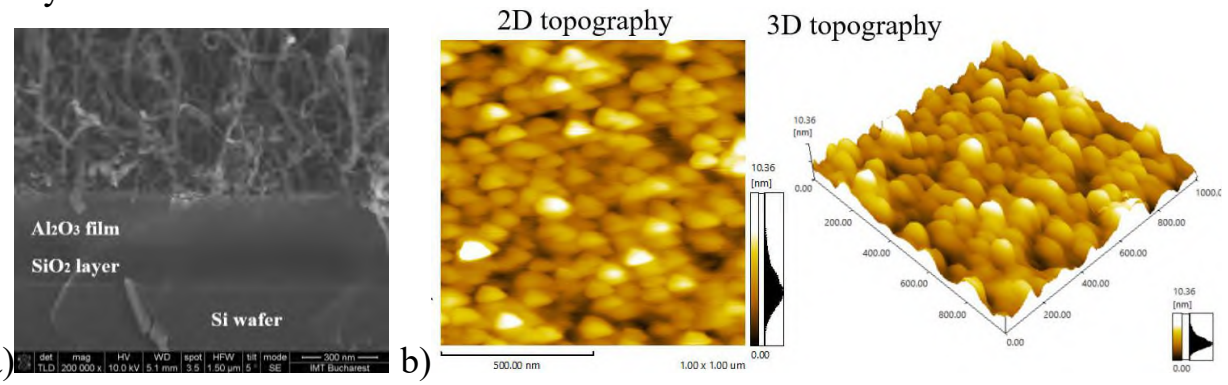


Figure 14. (a) Cross-sectional SEM image of CNTs synthesized on the $Fe/Al_2O_3/SiO_2/Si$ structure and (b) AFM topography of the Al_2O_3 layer after nanotube detachment.

The structural dimensions of carbon nanotubes synthesized on the $Fe/Al_2O_3/SiO_2/Si$ heterostructure were comprehensively investigated using Raman spectroscopy and high-resolution SEM analyses. Theoretical diameters (0.93–1.43 nm) calculated from the radial breathing modes (RBM: 174, 227, and 268 cm^{-1}) recorded in the low-frequency region of the Raman spectra confirmed the presence of single-walled carbon nanotubes (SWCNTs) in the sample. Nanotube diameters directly measured from high-magnification SEM micrographs (3.6 and 4.1 nm for SWCNTs, and 5.1 nm for DWCNTs) showed good agreement with these results, experimentally substantiating the formation of ultra-thin, small-diameter nanotubes on the surface.

The selective growth of specifically small-diameter SWCNTs was attributed to the surface morphology of the intermediate Al_2O_3 buffer layer. AFM topographical analysis of the substrate surface revealed that thermal annealing induced the formation of a nanoscale relief (nanopits) on the Al_2O_3 surface, characterized by an average surface roughness of 1.061 nm, depths up to 10.353 nm, and lateral widths of 3–8 nm. These nanocavities spatially confine the free surface diffusion of the molten Fe catalyst at elevated temperatures, effectively suppressing mutual coalescence and thermal agglomeration. Consequently, the Fe nanoparticles retain ultra-small and stable dimensions, creating favorable catalytic conditions exclusively for the nucleation and growth of small-diameter single-walled carbon nanotubes.

CONCLUSION

1. High-precision deposition regimes for Fe nanolayers with a thickness of 2.4–28.5 nm were determined via electron-beam physical vapor deposition (EB-PVD) by controlling the emission current and evaporation time. It was confirmed that the major part of the layers consists of the cubic α-Fe metallic phase, while the surface is composed of passivated FeO and α-Fe_2O_3 oxide phases.
2. Thermal treatment drives the transformation of the continuous Fe layer into discrete nanoparticles via surface diffusion, establishing that the threshold temperature for complete disintegration of an 8 nm thick Fe layer is 550 °C. At 750 °C, the critical thickness window for the formation of discrete catalytic centers across all buffer layers (SiO_2, Al_2O_3/SiO_2, TiO_2/SiO_2) lies within 3–12 nm, whereas complete disintegration is suppressed in layers with a thickness of 20 nm and above.
3. In the $Al_2O_3(120\ \text{nm})/SiO_2(160\ \text{nm})/Si$ system (700 °C, 5 min), a linear relationship $d \approx k \cdot h$ was established between the average nanoparticle diameter (d) and the initial Fe layer thickness (h), with the empirical coefficient k ranging within 4–6. Extending the thermal treatment time from 5 to 15 minutes was found to increase the average nanoparticle diameter from 32 nm to 85 nm and decrease the surface density from 83 to 23 particles/μm^2 due to coalescence and Ostwald ripening processes.
4. The morphological characteristics of Fe nanoparticles were shown to depend on the structural nature of the substrate and the buffer layer. On amorphous SiO_2 and Al_2O_3/SiO_2 surfaces, smaller quasi-spherical particles with high density are formed, which are 2.4 and 2.0 times smaller, as well as 14 and 3.4 times denser, respectively, compared to faceted particles on anisotropic crystal surfaces.
5. During carbon nanotube (CNT) nucleation, uncontrolled decomposition and increased concentration of hydrocarbon gas lead to the diffusion of C atoms into the catalyst particle, resulting in the formation of the Fe_3C phase, which blocks active sites and causes deactivation of the catalyst.
6. Strong interactions between defects on the CNT wall and the catalyst were found to reduce the active portion of the catalyst, elucidating its role in CNT termination.
7. It was proven that CNTs synthesized on the $Fe(8\text{--}9\ \text{nm})/Al_2O_3(120\ \text{nm})/SiO_2(160\ \text{nm})/Si$ structure exhibit a degree of perfection 19.2% higher than that of the TiO_2/SiO_2 system, 33.3% higher than the native SiO_2 layer, and 41.7% higher than the sapphire substrate, while also being 2.4–2.7 times superior in quality compared to low (550–650 °C) and high (800–850 °C) temperature regimes.
8. It was determined that nanoviscous/nanoscale pits ($R_a = 1.06$ nm) formed on the Al_2O_3 surface during thermal treatment restrict particle migration even in relatively thick (8–9 nm) Fe layers, enabling the synthesis of single-walled CNTs with a diameter of 0.93–1.43 nm. The developed $Fe(8\text{--}9\ \text{nm})/Al_2O_3(120\ \text{nm})/SiO_2(160\ \text{nm})/Si$ catalytic system is recommended as a promising technological foundation for obtaining high structural quality single- and multi-walled CNTs, as well as for creating robust composite insulators, sensors, and nanoelectronic devices.

НАУЧНЫЙ СОВЕТ DSc.05/25.27.12.FM/T.09.01 ПО ПРИСУЖДЕНИЮ УЧЕНЫХ СТЕПЕНЕЙ ПРИ ИНСТИТУТЕ ИОННО-ПЛАЗМЕННЫХ И ЛАЗЕРНЫХ ТЕХНОЛОГИЙ

ИНСТИТУТ ИОННО-ПЛАЗМЕННЫХ И ЛАЗЕРНЫХ ТЕХНОЛОГИЙ

ИСМАТОВ АСКАР АНВАР УГЛИ

ПРОЦЕССЫ ФОРМИРОВАНИЯ НАНОЧАСТИЦ НА ИНТЕРФЕЙСЕ КАТАЛИЗАТОР–БУФЕРНЫЙ СЛОЙ И ИХ ВЛИЯНИЕ НА РОСТ УГЛЕРОДНЫХ НАНОТРУБОК

01.04.12- Физика и технология наноматериалов

АВТОРЕФЕРАТ ДИССЕРТАЦИИ ДОКТОРА ФИЛОСОФИИ (PHD) ПО ФИЗИКО-МАТЕМАТИЧЕСКИМ НАУКАМ

Ташкент – 2026

Тема диссертации доктора философии (PhD) по физико-математическим наукам зарегистрирована в Высшей аттестационной комиссии при Академии наук Республики Узбекистан за номером B2026.1.PhD/FM1481

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Научный руководитель:	**Ашуров Хатам Бахронович** доктор технических наук, профессор
Официальные оппоненты:	**Таджибаев Ильхом Иброхимович** доктор физико-математических наук, старший научный сотрудник, **Хакбердиев Эльшод Олмосович** доктор философии (PhD) по физико-математическим наукам, старший научный сотрудник,
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С диссертацией можно ознакомиться в Информационно-ресурсном центре Института ионно-плазменных и лазерных технологий (регистрационный №26/18), по адресу: 100125, г. Ташкент, ул. Дурмон йўли 33.Тел.: (+99871)-262-31-69.

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(протокол реестра №26/18 от «20» ноябрь 2026 г.)

Х.Б. Ашуров
Председатель Научного совета по присуждению учёных степеней, д.т.н., профессор

И.Р. Ядгаров
Учёный секретарь Научного совета по присуждению ученых степеней, д.ф.-м.н., профессор

У.К. Махманов
председатель научного семинара при Научном совете по присуждению ученых степеней, д.ф.-м.н., профессор.

ВВЕДЕНИЕ (аннотация диссертации на соискание ученой степени доктора философии (PhD))

Задачи исследования:

разработать режимы прецизионного контроля толщины тонких пленок Fe, осажденных методом электронно-лучевого физического осаждения из паровой фазы (EB-PVD) на буферные слои SiO_2, Al_2O_3 и TiO_2, путем варьирования времени испарения и тока эмиссии, а также определить элементный и фазовый состав пленок;

определить стадии и граничные условия перехода сплошной тонкой пленки Fe в систему дискретных наночастиц при термической обработке (процесс десмачивания);

дать количественную оценку влияния толщины тонкой пленки Fe, температуры и продолжительности термообработки на наноструктурах SiO_2, Al_2O_3 и TiO_2 на размер, поверхностную плотность и распределение наночастиц по размерам, а также установить эмпирическую зависимость между диаметром наночастиц и толщиной пленки;

исследовать взаимосвязь нуклеации и роста УНТ с параметрами каталитических частиц, включая механизмы роста и физико-химические причины деактивации (отравления) катализатора;

провести сравнительный анализ структурного качества и морфологии УНТ, синтезированных на различных каталитических системах ($Fe/SiO_2/Si$, $Fe/Al_2O_3/SiO_2/Si$, $Fe/TiO_2/SiO_2/Si$, Fe/сапфир), и определить оптимальную архитектуру интерфейса катализатор–буферный слой;

изучить процессы каталитической деактивации наночастиц Fe при высоких температурах вследствие межфазного взаимодействия с буферным слоем и выявить оптимальный температурный диапазон для синтеза УНТ.

Объект исследования. Наночастицы Fe, синтезированные на буферных слоях SiO_2, Al_2O_3, TiO_2 и сапфировых подложках, а также сформированные на их основе углеродные нанотрубки.

Научная новизна исследования:

впервые показано, что на аморфных поверхностях SiO_2 и Al_2O_3 формируются квазисферические частицы высокой плотности (≈ 28 нм, 302 част./мкм^2), тогда как на кристаллических поверхностях сапфира и TiO_2 образуются ограненные и сравнительно разреженные частицы (≈ 68 нм, 17 част./мкм^2 и ≈ 56 нм, 87 част./мкм^2 соответственно). При этом доказано, что вследствие неравномерного распределения и укрупнения частиц ограничение поверхностной диффузии атомов Fe на данных кристаллических подложках выражено слабее по сравнению с аморфным Al_2O_3;

для тонкой пленки Fe, осажденной методом электронно-лучевого PVD в системе Al_2O_3 (120 нм)/SiO_2 (160 нм)/Si и подвергнутой термической обработке при температуре 700 °C в течение 5 минут, установлена эмпирическая зависимость $d \approx k \cdot h$ между средним диаметром наночастиц

(d) и начальной толщиной пленки (h); широкий диапазон ($k \approx 2–10$), приводимый в литературе для систем на основе Fe, для данных условий **впервые** сужен до интервала $k \approx 4–6$ (при $k \approx 5$), что позволяет предварительно оценивать и контролировать размеры каталитических наночастиц исходя из начальной толщины пленки для направленного роста УНТ;

впервые проведен комплексный сравнительный анализ УНТ, синтезированных на буферных слоях SiO_2, Al_2O_3/SiO_2, TiO_2/SiO_2 и сапфировых подложках. Установлено, что УНТ, полученные на тандеме Al_2O_3 (120 нм)/SiO_2 (160 нм), по показателям структурного качества превосходят нанотрубки на TiO_2 — на 19,2 %, на естественном SiO_2 — на 33,3 %, и на сапфире — на 41,7 %;

впервые даже при использовании относительно толстых (8–9 нм) слоев Fe в структуре Al_2O_3 (120 нм)/SiO_2 (160 нм) успешно осуществлен синтез однослойных УНТ ($d = 0{,}9–1{,}1$ нм). Раскрыто, что благодаря нанорельефной шероховатости поверхности буферного слоя Al_2O_3 ($R_a = 1{,}06$ нм) и его сравнительно сильному взаимодействию с частицами Fe, данная архитектура является наиболее эффективным буферным тандемом при температуре 700 °C.

Внедрение результатов исследования. Полученные в ходе диссертационного исследования результаты были использованы в фундаментальном проекте № AL-7623031231 на тему «Синтез углеродных нанотрубок на основе димера циклопентадиенилжелезодикарбонила и изучение их физико-химических свойств», выполненном в 2024–2025 годах в Институте общей и неорганической химии Академии наук Республики Узбекистан на основе узбекско-туркменского научного сотрудничества, при выявленных оптимальных кинетических условиях формирования наночастиц Fe на поверхности буферных слоев (толщина слоя Fe 8–9 нм, 700 °C, 5 мин; средний диаметр 25–30 нм, плотность ≈ 300 частиц/μm^2 в системе Al_2O_3(120 нм)/SiO_2(160 нм)) (справка АН РУз № 4/1255-3131 от 4 ноября 2026 г.). Эти результаты решили проблему контроля размера частиц катализатора: была создана возможность синтеза высококачественных, отлично кристаллизованных одностенных углеродных нанотрубок на основе активных каталитических центров с жестко контролируемыми размерами и оптимизации их свойств.

Закономерности, полученные по синтезу нанотрубок методом CVD на гетероструктурах $Fe/Al_2O_3/SiO_2/Si$, $Fe/TiO_2/SiO_2/Si$ и $Fe/SiO_2/Si$ — в частности, влияние природы промежуточного слоя, температурного режима и продолжительности реакции на морфологию и качество наноструктур — были использованы в прикладном проекте на тему «Повышение эффективности высоковольтных изоляторов марок ИПП-10/1000-8А УХЛ1 и ЛК 70/10-3 УХЛ1 на основе кремнийорганического полимера», реализованном в 2024–2026 годах на совместном предприятии ООО «Арматура-Изолятор заводи (АИЗ)» (справка СП ООО «АИЗ» № 19 от 16 января 2026 г.). Данные результаты решили проблему обеспечения оптимальной концентрации и

равномерной дисперсии нанотрубок в композите изоляторов, в результате чего повысилась эксплуатационная стойкость высоковольтных изоляторов марок ИПП-10/1000-8А и ЛК 70/10-3 к факторам внешней среды и механическим нагрузкам.

Структура и объём диссертации. Диссертационная работа состоит из введения, четырех глав, заключения, списка использованной литературы и приложений. Основной текст работы изложен на 117 страницах и содержит 56 рисунков и 7 таблиц.

E'LON QILINGAN ISHLAR RO'YXATI
СПИСОК ОПУБЛИКОВАННЫХ РАБОТ
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