MAGYAR TUDOMÁNYOS AKADÉMIA

KARBON ALAPÚ NANOSTRUKTÚRÁK MORFOLÓGIAI JELLEMZÉSE KÉPELEMZÉS SEGÍTSÉGÉVEL

DOKTORI ÉRTEKEZÉS TÉZISEI

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BEVEZETÉS ÉS AZ ÉRTEKEZÉS CÉLKITŰZÉSEI

A heterogén nanostruktúrák jellemzésének ma ismert leghatékonyabb eszköze a nagyfelbontású elektronmikroszkópia, az eredmények elemzése azonban meglehetősen idő- és munkaigényes folyamat. A karbon alapú nanostruktúrák különösen érdekesek lettek az utóbbi évtizedekben, hisz pl. a grafit, a szintetikus gyémánt, a fullerének, vagy legújabban a grafén, ill. a rájuk jellemző különleges tulajdonságok még a napi híradásokban is egyre gyakrabban szerepelnek. Az említettek mellett talán kevésbé izgalmasan hangzó anyag a korom, mely az égési folyamatok során mellék-, ill. egyes esetekben fő termékeként keletkező karbon alapú anyag. Számos felhasználási területe mellett szennyezőként sem hanyagolható el, ezért is foglalkoztatja a kutatókat évtizedek óta világszerte.

Az alapvetően karbonatomokból felépülő koromrészecskék szinte mindenhol jelen vannak a légkörben, hiszen a repülőgépek turbináiban, a dízel és egyéb belsőégésű motorokban, számos ipari és lakossági tüzelőberendezésben, valamint természetben zajló égési folyamatok generálják azt a aeroszolt, amit koromként ismerünk. A korom parányi belélegezhető részecskéi mélyre hatolhatnak a tüdőbe és a korom, illetve a hozzá kapcsolódó többgyűrűs aromás szénhidrogének (az ún. PAH-ok) rákkeltő hatása régóta ismert és széleskörűen dokumentált. A korom további környezetszennyező hatása például a látótávolság csökkenése, vagy a szmog képződése.

A HRTEM képekből a fizikai értelemben tartalmas, megbízható, pontos és statisztikailag robusztus adatok kinyerése nem könnyű művelet. Az adatok kinyerését különböző jelenségek akadályozzák: pl. egymást fedő szerkezetek, változó fókusz, kontraszt és megvilágítási szintek valamint a képekben jelenlevő zaj. Az egyértelmű jellemzés érdekében ugyanakkor jól meghatározott és mindenre kiterjedő szerkezeti paraméterek szükségesek a számszerűsítéshez. Az általában megfigyelhető látómező és az egyetlen elektronmikroszkópos felvételből kinyerhető szerkezeti információ többnyire elégtelen mennyisége további problémát jelent a megbízható statisztikai leíráshoz.

Ebben az értekezésben a következő kérdésekre keresek választ:

- Milyen módszerrel lehet a már kifejlesztett és publikált szerkezeti deskriptorokat egyesíteni és hogyan lehet a szerkezeti paramétereket a lehető legpontosabban mérni?

- Miképpen lehet az általam korábban fejlesztett algoritmus szolgáltatta adatmennyiségnél nagyságrendekkel több adatra épülő, robusztus képelemző módszert fejleszteni?
- Létezik-e olyan módszer, ami figyelmen kívül hagyja a kép bizonytalan, (határozott struktúrával nem rendelkező) részeit? Lehet-e hibatűrő (pl. a képzajra érzéketlen) algoritmust implementálni?
- Kimutatható-e képelemzéssel a karbon-szerkezetek alapanyagtól való függése? Hogyan változik a korom nanoszerkezete lamináris diffúz lángban? Kimutatható-e függés a láng méretétől?

A kifejlesztendő algoritmus tesztelésének tervezett lépései:

- Validálás pontosan ismert geometriájú szintetikusan generált képekkel,
- Összehasonlítás korábban már vizsgált – jól számszerűsíthető – grafit minták elemzései eredményeivel.

A módszer használhatóságát demonstrálni kívánom nehezen kvantifikálható amorf korommintákon is.

ÚJ TUDOMÁNYOS EREDMÉNYEK

Továbbfejlesztettem a korábban kidolgozott – nagyfelbontású transzmissziós elektronmikroszkóp és számítógépes képelemzés összekapcsolásán alapuló – koromszerkezetet számszerűsítő módszert, és több példán demonstráltam a módszer gyakorlati alkalmazhatóságát. A kialakított algoritmus révén lehetővé válik a hasonló szerkezetű karbonalapú anyagok (korom, műkorom, grafit, stb.) szerkezetének kvantitatív leírása, a mikro és nanoszerkezetbeli különbségek számszerűsítése. A módszer kidolgozásakor előrevetítettem, hogy a kvantitatív szerkezet meghatározás megteremtheti az alapot a légköri korom minták morfológiai elven történő eredet-meghatározásához, vagy forrás-hozzárendeléséhez. A koromszerkezet egyértelmű leírhatósága elősegítheti a tüzelési folyamatok optimalizálását, továbbá eszközt nyújthat az emisszió monitorozására és a forrás-hozzárendelésre. Ennek eredményeként a kibocsátók „megnevezése” és a szükséges intézkedések megtétele csökkentheti a tüzelési eredetű légköri szilárd szennyezők okozta káros hatásokat.

1. Új – szegmentáción alapuló - képfeldolgozási algoritmust fejlesztettem ki amorfnak tekinthető korom nanoszintű morfológiája elemzésére. A módszer robusztusságát összehasonlító elemzésekkel igazoltam. Az algoritmus az alábbi lépésekből áll:
    a) Fourier transzformáció
    b) Gauss-féle low-pass szűrés a frekvencia doménen, majd inverz Fourier transzformáció
    c) Intenzitás homogenizálása hisztogram műveletekkel
    d) Lokális intenzitás minimumok keresése és megjelölése
    e) Szegmentált maszkok előállítása a Vincent-Soille watershed algoritmussal, a források a megjelölt lokális minimumpontok
    f) A szegmentált maszkok kivonása az eredeti, frekvenciaszűrt képből
    g) A szegmentált síkok címkézése a hatásterületek határain
    h) A címkézett szegmensek lokális binarizációja
    i) A bináris kép rekonstruálása a szegmensek megfelelő pozícióra való rajzolásával
    j) Utószűrés
2. Kifejlesztettem a Gábor-szűrők alkalmazására alapuló morfológia meghatározására szolgáló módszert. Ez különbözik minden korábban publikált megközelítéstől és képes arra, hogy az eddig ismert „standard” módszerekhez viszonyítva nagyságrendekkel több szerkezeti információt nyerjen ki egyetlen képből. A módszer gyakorlatilag érzéketlen a zajra, a fázisinverzió jelenségére és az elméletileg lehetséges legkisebb lokalizációs bizonytalanságot mutatja. Az algoritmus lépései a következők:
    a) A releváns hullámhosszsáv megadása manuálisan, vagy automatikusan az energia spektrum alapján.
    b) A Gábor-szűrő paraméterek meghatározása és ezek alapján a szűrőkészlet megszerkesztése.
    c) A kép szűrése a készletet alkotó minden egyes szűrő alkalmazásával, és a diszkrét válaszok rögzítése minden egyes pixelnél.
    d) A folytonos interpolált válasz felületmaximumaihoz tartozó frekvenciák megkeresése és rögzítése.

e) A szerkezeti paraméterek (θ, λ és μ) meghatározása és tárolása, ugyancsak pixelenként.

3. A standard módszerektől eltérően, a kifejlesztett algoritmus natív képfelbontásnál biztosít szerkezeti információt, azaz a kép minden egyes pixelje szerkezeti paraméterek egy sorát eredményezi. A módszer használhatóságát mesterségesen létrehozott képeken, szintetikus grafitok valódi mikroszkópos felvételein és amorf korom valódi mikroszkópos felvételein igazoltam. Bizonyítottam, hogy a módszer a standard algoritmusokkal elérhető eredményekkel megegyező információt biztosít grafitos szerkezetek esetében, azonban olyan esetekben is képes nagy megbízhatóságú adatokat szolgáltatni, ahol a standard technikák erre képtelenek - különösen az amorf korom mintáinak az esetében.

4. A különböző alapanyagból készített szintetikus grafit minták szerkezetét vizsgálva bizonyítottam, hogy az előállított karbonszerkezet függ az alapanyag kémiai összetételétől. Képelemzéssel igazoltam a rácssíktávolság multimodális jellegét.

5. Elsőként közöltem eredményeket korom mikroszerkezet fejlődésének HRTEM alapú vizsgálatáról lamináris diffúz lángban. Kimutattam a rácssík-távolság enyhe csökkenését, amint a korom átvonul a növekedési régióból a nagyhőmérsékletű oxidációs zónába. Nagyobb, instabil diffúz láng, illetve egy nagy kerozin tartálytűz aktív lángjából vett korommintával összehasonlítva azt találtam, hogy a nagyobb lángokban a rácssík-távolság is nagyobb, mint a referencia lángból vett mintáé.

TUDOMÁNYOS EREDMÉNYEK HASZNOSULÁSA

Az adatpontok nagyobb száma a szerkezet pontosabb és megbízhatóbb statisztikai értékelését jelenti. Annak, hogy egyetlen felvételből a legnagyobb mennyiségű szerkezeti információt tudjuk kinyerni, szignifikáns jelentősége van az olyan esetekben, ahol a minták vagy a mikroszkópos felvételek csak korlátozottan állnak rendelkezésünkre. Mivel a koromból való mintavétel tipikusan bőséges mennyiségű koromszemcsét eredményez, nem jellemző a túl alacsony mintaszám, mint korlátozó tényező. A koromrészecskék heterogenitásától függően azonban meglehetősen időigényes folyamat az elegendő számú olyan mikroszkópos felvétel készítése és elemzése, amelyek elegendőek robosztus statisztikai feldolgozást tesznek lehetővé. Az ilyen esetekben az, hogy képesek vagyunk minden egyes mikroszkópos felvételből annyi információt kinyerni, amennyit csak lehetséges, nem csupán az eredmények megbízhatóságát növeli, hanem gyakorlati és gazdasági jelentőséggel is bír.

A képelemzés szolgáltatta adatok lehetőséget nyújtanak egyrészt az égés során végbemenő folyamatok jobb megértéséhez, másrészt megteremtik a lehetőséget a légköri korom, mint szennyező forrásának azonosítására, ezáltal pedig a konkrét kibocsátás-csökkentés környezetvédelmi, illetve ezen keresztül egészségügyi hatású beavatkozásokra.

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