

Controlling the structure and morphology of ZnS nanoparticles by manipulating the temperature profile

CHAN GI LEE • AIST • change-ri@aist.go.jp

YUSUKE NAKAMURA • Kyushu University • md-y-nakamura@mms.kyushu-u.ac.jp

HIROYUKI NAKAMURA • AIST • nakamura-hiroyuki@aist.go.jp

MASATO UEHARA • AIST • m.uehara@aist.go.jp

HIDEAKI MAEDA • AIST, Kyushu University, CREST • maeda-h@aist.go.jp

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We propose a method to control the kinetics by controlling the reaction condition stringently with time. One-pot temperature triggered reaction system was employed with identical raw material solutions of ZnS nanoparticles and several patterns of heating profile to show their effects and usefulness during the preparation process were applied. ZnS raw materials were heated up by two type heating rate profile (0.01 °C/sec, 500 °C/sec) to achieve the desired temperature. In high heating rate (500 °C/sec), zinc blend (ZB) phase spherical nanoparticles were obtained above 125 °C. Wurtzite (WZ) phase ZnS nanorods were synthesized under a low heating rate condition (0.01 °C/sec). Furthermore, the crystal phase of the final particles is determined by the initial temperature during particle generation.

Keywords: phase and morphology control, temperature profile, ZnS nanoparticle

Introduction

Potential applications of nanoparticles are expected for various fields such as electronics, energy, and biology. Generally, the physical and chemical properties of nanoparticles are dependent on the structure (e.g. defects, composite structures, and crystal phases) and morphology (e.g. size and shapes of particles) [1, 2]. Therefore, structure and morphology control of nanoparticles is an important issue, and many reports on the synthesis of metal and semiconductor nanoparticles have been reported [1, 2]. Generally for a nanoparticle synthesis by chemical solution process, a reaction condition, which is determined by the type and concentration of each chemical reagents, reaction temperature and time etc., are selected to control the reaction thermodynamics and kinetics. Because the nanoparticle preparation process basically treats a non-equilibrium states, apart from the equilibrium control of reaction, kinetic control of processes such as nucleation, growth, aggregation, and ripening are important [1–3]. The importance of kinetic control is recognized by many researchers, especially for particle size and distribution. Timing and rate of nucleation and growth that is a result of comprehensive growth mechanism that is influenced by surface reaction rate and monomer feeding rate are said to strongly affects the particle size distribution [4–6] and particle shape [1, 2]. Moreover, in recent years, some studies revealed the effect of kinetic control on crystal phase, defect generation and doping concentration of nanoparticles [7–16]. Generally, surface energy and volume energy determine the total free energy of nanoparticles. Therefore, the factors to determine those energies (e.x. particle diameter, sort and area of crystal face, adsorption and desorption of capping agents (i.e. surface ligands)) are affected by several kinetics including nucleation and growth kinetics and also by adsorption-desorption kinetics of capping agents. As such, the kinetic control can be a useful factor for colloidal

Dr. Chan Gi LEE

is Post-Doctoral Fellow in Micro-space Chemistry Solution team, Measurement Solution Research Center (MSRC), National Institute of Advanced Industrial Science and Technology (AIST), Japan. He received doctoral degree at Kyushu University in 2009.

Yusuke NAKAMURA

is graduate student in Kyushu University and he received the master degree in March, 2011.

Dr. Hiroyuki NAKAMURA

actually is senior researcher in Micro-space Chemistry Solution Team, MSRC, AIST, Japan. After he received his doctoral degree in 1994 in engineering from Kyushu University, he worked for AIST and Shimane University (1997–1999), and again AIST from 2000. His current research field is micro-space chemistry for inorganic nanomaterials.

Dr. Masato UEHARA

actually is a researcher in Micro-space Chemistry Solution Team, MSRC, AIST, Japan. After he received his doctor degree at Kyushu University in 1998, he worked for Kyushu University, and he joined AIST from 2002. His research focuses on using electron microscope and X-ray analysis to investigate and develop nanocrystal materials.

He has investigated the phase stability and morphology of ZnS nanocrystals in recent years.

Prof. Dr. Hideaki MAEDA

actually is leader of Micro-Space Chemistry Solution Team, MSRC, National Institute of Advanced Industrial Science and Technology (AIST), Japan, Professor of Kyushu University, and Research Director of CREST, JST. After he received his master degree from Kyushu University in 1989, he worked for Mitsui mining company LTD., Kyushu University and joined AIST from 1999. He has got engineering degree at Kyushu University in 1994. His current research field is micro-space chemistry.

nanoparticle synthesis procedure. The full understanding and exploitation of such kinetic effects will help more in controlling the production of nanoparticles, and furthermore, it may help realize a new process, e.g. preparation of various structures and morphologies (i.e. different properties) of nanoparticles from a handful of types of reaction systems. There are two types of methods for kinetic control. One is to control the reaction kinetics by reaction conditions [5, 8, 9, 11, 12, 16], and the other is to control the reaction conditions stringently with time [7, 10, 13, 14]. In the current state, the former is dominant because it is rather difficult to control the reaction condition with time in a quantitative manner; however, it is possible to find some reports for the latter case. For example, Manna et al has reported on kinetic effects on shape of CdSe nanocrystals. They controlled branching and phase of CdSe nanocrystals by “rapid” and “slow” injection of CdSe raw material solution with proper timing [13]. Also, Cozzoli et al. reported phase and shape control of ZnSe nanocrystals except control over ligands. The injection of large volumes of stock solution yielded spherical particles in the exclusive cubic ZB structure, whereas slow, dropwise addition of the same stock solution promoted the formation of rodlike in the WZ phase [10]. However, from the viewpoint of controllability of kinetics, quantitative control of mixing is not easy – a partially high concentration and temperature fluctuations that can be generated during raw material injection may give unwanted effects. Puentes et al.

showed that the heating time of the reaction solution can affect the crystal phase of Co [14]. Therefore, in this study, we employed a simple temperature triggered reaction. Here it should be noted that a microreactor, which is a small flow type chemical reactor whose representative size is less than 1 mm, is a very useful tool for precise temperature control with time [17]. The reactor can heat room temperature reaction solution up to reaction temperature (e.g. 200 °C) within ca. 0.5 s, in a homogeneous manner [18].

In this study, we tried to control the phase and morphology of ZnS. ZnS has a wide optical band gap (3.6 eV), and a promising material for novel applications including light-emitting diodes (LEDs), electroluminescence, and sensors, etc. [19]. ZnS has two representative polymorphs, i.e. cubic ZB structure and hexagonal WZ structure. For bulk system, the ZB at low-temperature phase is more stable while the WZ at high-temperature form polymorphs at around 1023 °C [20]. However, DFT calculation showed that the phase stability also depends on particle size (diameter) and WZ phase is more stable than ZB phase when the particle size was smaller than 4 nm [21], as the result of high surface/volume ratio. Furthermore, several researchers have reported that the composition of ligands and solvents in the reaction solution affect the crystal phase, because these factors influence the surface energy of particles [22–24]. As such, the crystal phase of the ZnS nanoparticles is considered to be strongly affected by the size and surface energy of nanoparticles. Therefore, the stability of the phase can be affected by the particle size (i.e. surface/volume ratio) and shape (i.e. crystal habit) as the result of growth process (i.e. growth mechanism), and reaction environment around the nanoparticles (i.e. thermodynamical equilibrium including surface state of the particle). Therefore, many researchers report on the phase and shape control of ZnS nanoparticle by the solution process [23–25].

From this point of view, we selected ZnS nanoparticles as a model material, and tried to control the crystal phase and morphology by kinetic control. For simplicity, one-pot temperature triggered reaction system was employed with identical raw material solutions, and applied a few patterns of heating profile to show the effects and usefulness of the heating pattern during the preparation process.

Experimental procedure

Zinc iodide (ZnI_2 , 99.999%) and sulfur (S) powder were provided by Aldrich. Oleylamine and 1-octadecene (reagent grade) were purchased from Acros Organic and Wako Pure Chemicals, respectively. All solvents were vacuum distilled before use. ZnS nanocrystals were prepared by a solution process called “organo-metallic route” [26], ZnI_2 was dissolved in oleylamine (0.03 mol/L) at room temperature, and S powder was dissolved in octadecene at 150 °C (0.15 mol/L). After cooling the solution to room temperature, these solutions were mixed and used as a raw material for ZnS nanocrystals. ZnS nanocrystals were synthesized with several pattern temperature profiles. For slow heating rate, the raw material solution for ZnS was heated in oil bath, immediately after mixing Zn and S source solution in a glass vessel. The heating rate was controlled

using PID (proportional–integral–derivative) controller. For rapid heating, a glass capillary with an internal diameter (ID) of 200 μm was used to heat the raw material solution and collected in a glass vessel for further heating. A heat transfer calculation was used based on the expectation that in room temperature the solution can be heated within 0.4 s up to 200 °C (500 °C/sec) for the current type of microreactor [18].

The product nanocrystals were washed with ethanol and redispersed in hexane. ZnS nanocrystals were characterized by X-ray diffraction (XRD, RINT-TTR; Rigaku) and scanning transmission electron microscope (STEM, S-5200; Hitachi High-Technology) to determine the crystal phase and morphologies. In addition, the average particle sizes and rod lengths were calculated by analysis of SEM images (Quick Grain).

Results and discussion

At first, two heating profiles were applied for ZnS nanoparticle synthesis: profile A-heating with heat-up from room temperature to 200 °C by using a heating rate of 0.01 °C/s and maintenance at 200 °C for 1h, and profile B-heating with an increase in temperature up to 200 °C by heating rate of 500 °C/s and maintenance at 200 °C for 1h. In order to observe the development of particle shape and phase, an aliquot of the sample was purified for STEM and XRD analysis. The schematics of applied temperature profile and morphology for each sampling point is shown in Fig. 1. XRD diffraction pattern for each sampling point is shown in Fig. 2. With the low heating rate (0.01 °C/sec) condition (profile A: Fig. 1.) the product yield (PY) at 100 °C was 60% and it reached ~100% at 125 °C. At 100 °C, the produced particles were very small (<~2 nm) and the morphology was not clear. However, at 125 °C, the morphology was rather ellipsoidal, whose short axis was about 3 nm and long axis was 5 nm. With an increase in temperature up to 175 °C, the length of the particle increased to 7 nm (150 °C) and 8 nm (175 °C) without showing obvious change in short axis (3 nm). On the other hand, the crystal phase at 100 °C was not clear (Fig. 2. (a)), but at 125 °C the hexagonal WZ structure started to become visible and completely dominated at 175 °C. These results indicate that ZnS generation by chemical reaction is already completed at 125 °C, and the c axis of wurtzite (WZ) ZnS grows with temperature with maintenance of constant width. This result suggests an oriented attachment of WZ ZnS nanoparticles (and rods) wherein they serve as building blocks [27]. On the other hand, from 175 °C to 200 °C, the growth became isotropic with the short and long axis increasing to 4 nm and 10 nm, respectively. The following 1h aging showed no difference in the average length and width, but based on the STEM image, their size distribution appears to increase by aging. At 200 °C, the WZ peak became weaker than that at 175 °C and less obvious after 1h of aging. The isotropic growth of rod like ZnS under equilibrium yield implies that the growth is caused by Ostwald ripening. The reason for the change in the growth mechanism is not clear at present but can probably be caused by enhancement of the material transfer rate (i.e. dissolution and re-deposition rate) due to Ostwald ripening. It should be noted that the increase in zinc blende (ZB) phase at 200 °C might show that ZB phase is more preferred at this

temperature for the current reaction system. In the case of profile B, after the temperature reached 200 °C within 1 s, the product yield increased with aging time; 15% (5 s), 40% (10 s), 60% (20 s) and 85% (60 s). The morphology of the particles were hardly observable by STEM before 10 s, but at 20 s (PY~60%), small isotropic particles of ~2 nm in diameter were observed, and characterized as ZB structure by XRD. Further aging made the particles grow to 3.5 nm (10 min) and 4.0 nm (60 min) without any change in isotropic morphology and crystal phase (ZB) under an equilibrium product yield. The crystal phase for the high heating rate (profile B) was ZB from the early stage and was different in comparison to the WZ observed for the low heating rate (profile A). This result agrees with the former results which indicate higher stability of ZB at 200 °C.

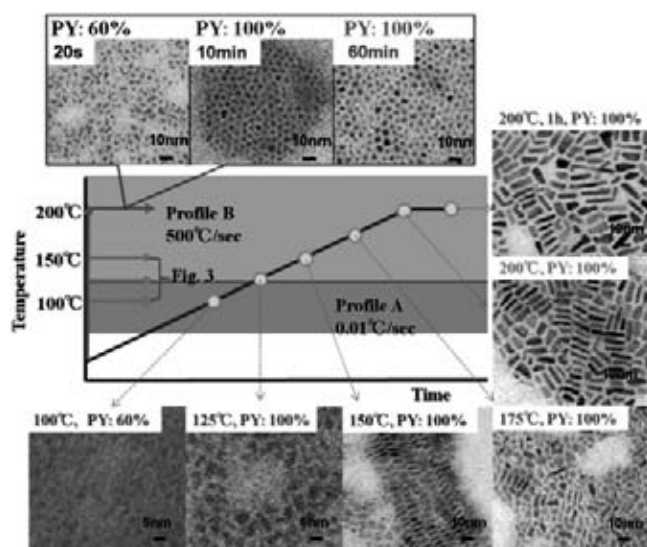


Fig. 1. Schematic diagram of temperature profile and SEM images of ZnS nanoparticles

1. ábra A hőmérsékletprofil és a ZnS nanorészecskék pásztázó elektronmikroszkópos (SEM) felvétele

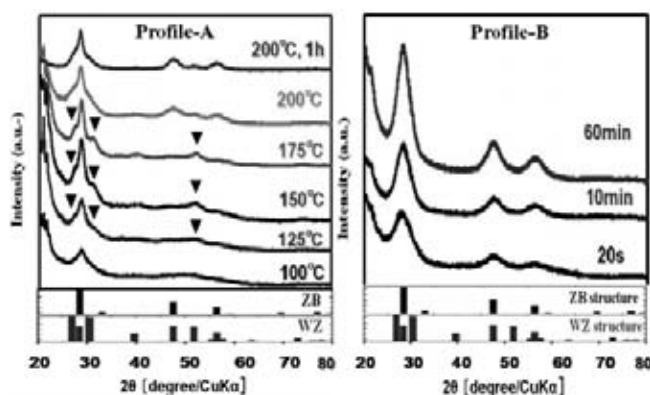


Fig. 2. Typical XRD pattern of ZnS nanoparticles; (a) high heating rate (500 °C/sec), (b) low heating rate (0.01 °C/sec).

2. ábra ZnS nanorészecskék jellemző röntgen-diffraktogramjai (a) fűtési sebesség 500 °C/s (b) fűtési sebesség 0,01 °C/s

The above results clearly show the effects of temperature profile on the phase and morphology of ZnS nanoparticles. The low heating rate caused the formation of WZ type ZnS at 125 °C then anisotropically grew by oriented attachment and finally formed rod like particle at 200 °C, while the particles

generated at 200 °C were isotropic ZB nanoparticles. Following these results, to investigate and clarify the stable structure for temperature between 100 °C to 200 °C, we further modified the temperature profile and performed another experiment with the following conditions: the raw material solution was heated up to 100, 125, and 150 °C with high heating rate (500 °C/sec) and kept at each respective temperature for 1h. The results are shown in Fig. 3. At 100 °C, very thin WZ ZnS nanorods (width ~1.5 nm) which resemble magic size nano wires recently reported by Den et.al. [29] were generated, whereas at 150 °C, isotropic ZB nanoparticles were obtained. At 125 °C, the product had a 2 nm diameter and appeared rather isotropic, but the phase was a mixture of WZ and ZB structures. From these results, it can be concluded that ZB was preferably generated above 125 °C, whereas WZ was favored below 125 °C. The difference in the crystal phase in the different generation temperatures could be the main reason for the temperature profile effects shown in Fig.1.

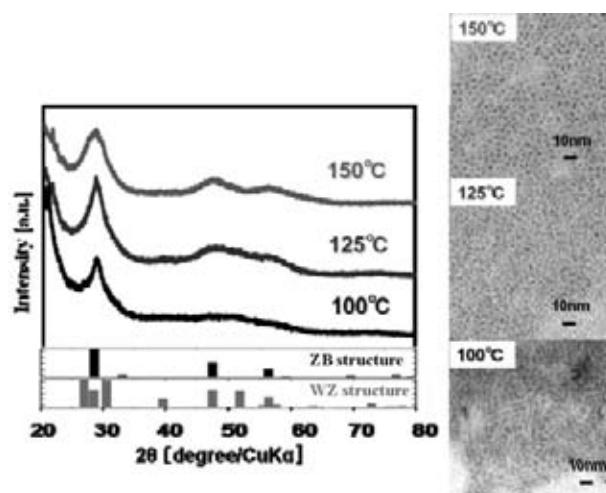


Fig. 3. XRD pattern and SEM images of ZnS nanoparticles 100, 125, and 150 °C with high heating rate (500 °C/sec)

3. ábra ZnS nanorészecskék röntgen-diffraktogramjai és SEM felvételei 100, 125 és 150 °C-on, 500 °C/s fűtési sebesség mellett

Finally, we tried to use the heating profile control to skip or enhance the critical temperature region for WZ particle. To enhance the WZ structure, the reaction solution was heated at a rate of 0.005 °C/s to reach 100 °C and kept at this temperature. As shown in Fig. 4., the product had clear WZ phase and thicker ZnS nanorod with 3 nm width and 20 nm length. The width and length were much larger than those subjected to 1h aging (Fig. 3.) regardless of the constant product yield, suggesting that ripening process proceed to enhance the WZ structure at this temperature. In contrary, the reaction mixture was heated at a rate of 500 °C/sec to reach 200 °C (Fig. 1. profile-B) and kept at this temperature for 20 s, then quenched to 75 °C and kept for 24h as shown in Fig. 5. This profile skips the temperature for WZ structure generation but aged at the temperature for WZ structure. The resultant particles had ZB structure with 3 nm that indicate isotropic ripening growth. The results show that ZB structure particles are favored over Ostwald ripening, and the initial heating profile actually determines the fate of the particles for this temperature. Consequently, temperature profile considering the critical temperature region is important

to control the phase and morphology of ZnS nanocrystals by the current method.

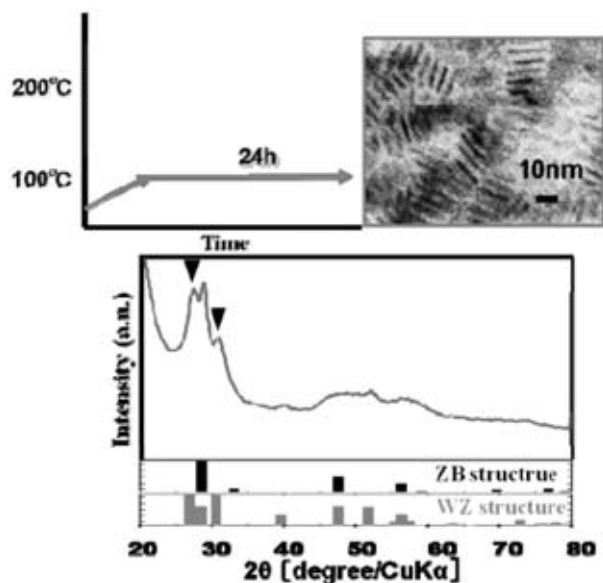


Fig. 4. XRD pattern and SEM images of ZnS nanoparticles at 100°C 24h (heating rate: 0.005°C/sec)

4. ábra ZnS nanorészecskék röntgen-diffraktogramjai és SEM felvételei 100°C-on, 24 órás kezeléskor, 0.005°C/s fűtési sebesség mellett

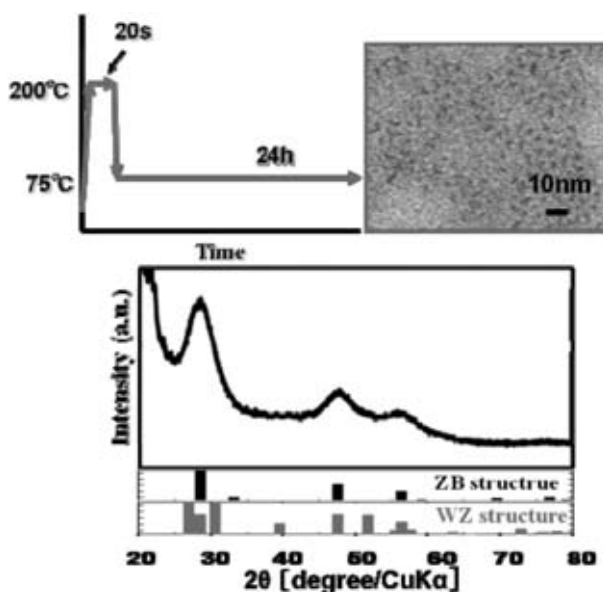


Fig. 5. XRD pattern and SEM images of ZnS nanoparticles at 75°C, 24h (heating rate: 500°C/sec to 200°C)

5. ábra ZnS nanorészecskék röntgen-diffraktogramjai és SEM felvételei 75°C-on, 24 órás kezeléskor, 500°C/s fűtési sebesség mellett

Conclusion

In this study, ZnS nanocrystals were synthesized from identical raw material solutions, and the effects of temperature profile on the resultant phase and morphology of ZnS nanocrystals were investigated. It was possible to obtain a rod like ZnS particles by slow heating (0.01°C/s) up to 200°C, whereas a fast heating rate (500°C/s) formed spherical nanoparticles. Particle generation and growth during those processes revealed that wurtzite (WZ) ZnS nanoparticles were generated

below 125°C for the slow heating rate and undergo oriented attachment to generate rod like particles, whereas the fast heating rate generates zinc blende (ZB) type ZnS nanoparticles after reaching 200°C, showing the clear effects of particle generation temperature on the crystal phase. A series of fast heating rates up to various temperatures revealed the effects of generation temperature and showed that the marginal line for ZB type and WZ type particle generation was around 125°C. Further experiment on aging for a long period of time for WZ generation region enhanced the WZ structure to give thick rod like particles, whereas skipping WZ generation region by rapid heating presented ripening of spherical ZB nanoparticles at low temperature aging. Consequently, the series of simple experiments successfully showed the importance of temperature profile for shape and phase control even in cases of using the same starting solution. In our additional experiments, we also tried to control the phase and shape of ZnSe nanocrystals via temperature profile by using microreactor which made it possible to realize more precise heating profile control. The results will be reported in our next publication.

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Cink-szulfid nanorészecskék szerkezetének és morfológiájának szabályozása a hőmérsékletprofil változtatásával

Módszert javasolunk reakciók időbeli lefutásának szabályozására a reakciókörülmények szigorú időbeli beállításával. ZnS nanorészecskéket tartalmazó, azonos összetételű oldatokban, megfelelő hőmérsékleten beindított szakaszos reakciók esetén tanulmányoztuk a különböző hőmérsékletprofilok hatását a lejátszódó folyamatokra. A ZnS kiindulási anyagot két különböző fűtési sebesség (0,01 °C/s és 500 °C/s) mellett melegítettük fel a kívánt hőmérsékletre. Nagy fűtési sebesség-nél cink-elegyfázisú (ZB), gömbszerű nanorészecskékből álló anyagot kaptunk 125 °C felett. Kis hűtési sebességnél Würzitet tartalmazó (WZ) ZnS nanorudak keletkeztek. Megállapítottuk továbbá, hogy a végtermékek fázisviszonyait a részecskék előállításakor alkalmazott kiindulási hőmérséklet határozza meg.

Kulcsszavak: fázisösszetétel és morfológia szabályozása, hőmérsékletprofil, ZnS nanorészecske

FOLYÓIRATSZEMLE

Isoliertechnik

2011. szeptember

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- Aktuális hírek a Korff, a Lindner, az Isover, a Dr. Starck & Co. GmbH, az Armacell, a Rockwool és Kaefer cégektől
- ISO'12, Európa legnagyobb szigeteléstéchnikai szakkiállítás Kölnben, 2012. május 10–11-én

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Regenhart Péter

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2011. március, pp. 11-14.

Stuart Hakes: **Hogyan javítja az űrkomponál alkalmazott bevonási technológia az üveglvasztó kemencék boltozatának emissziós tényezőjét**

Az üveglvasztó kemencék energia-megtakarításának új módszerét mutatja be a cikk szerzője, mely a közelmúltban nyert ipari alkalmazást.

Egy licencadási program keretében lehetővé vált, hogy a NASA által kifejlesztett, és az űrkomponál külső szigeteléséhez nano-részecskéket felhasználó bevonatokat az üveglpar is átvehesse. Ehhez előzően olyan kötőrendszereket kellett kifejleszteni, mellyel a bevonat a kemence tűzálló falzatának az üveggel nem érintkező felületére felhordható. A bevonat igazi haszna, hogy emissziós tényezője magas hőmérsékleten megközelíti az elméleti (fekete test) értéket. Míg a kemence tűzálló falzatának emissziós tényezője a 0,4-0,6 közötti tartományban van az olvasztás hőmérsékletén, addig az Emissshield márkanévű bevonat kb. 0,9. Ez azt jelenti, hogy a bevonat által elnyelt hő mintegy 90%-át visszasugározza a hidegebb üveglvadéokra.

A bevonatot kereskedelmileg 2006-ban vezette be a NARCO a FIC (UK) céggel együttműködésben, és mára már számos, elsősorban multinacionális cég vette alkalmazásba. A bevonat viszonylag egyszerűen, felszórással (szórópisztollyal) hordható fel a hideg kemencék belső felületére. Meg kell jegyezni, hogy a bevonatot nem célszerű öntött AZS blokkokra alkalmazni, mivel azok magas hőmérsékleten üveges fázist „izzadnak” ki magukból, ami jelentősen csökkenti a bevonat hatékonyságát. A cikk szerinti 28 alkalmazás fele un. „oxy-fuel”, míg másik fele hagyományos gáztüzelésű kemencénél került bevezetésre különböző üvegtípusoknál: a palacküvegtől a vízüvegen át a börtartalmú üvegig.

Az Emissshield szélesebb körű kipróbálása újabban kiterjed a munkakádák, fűtött csatornák és a gyártó szerszámok területére is.

A bevonat komplex gazdasági és környezeti előnyökkel bír. A cikk szerinti 28 gyakorlati alkalmazás és több éves üzemeltetés után elmondható, hogy 10% földgáz-megtakarítás is elérhető a kemencék méretétől függően. Emellett természetesen arányosan csökken a kemencék CO₂, SO_x és NO_x kibocsátása is.

Lipták György