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Soft Processing for Sustainable Society – Possibility of Water-Based Processing for Advanced Inorganic Materials

Growing Integration Layer [GIL] Strategy: Direct Fabrication of Compositionally, Structurally and Functionally Graded Ceramic Coatings and/or Films from Mother Materials in Solution

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(predavanja će se održati u dva bloka po 45 minuta, s pauzom od 15 minuta)



Profesor Yoshimura svjetski je poznati stručnjak za keramičke materijale i njihovo dobivanje tzv. "mekanim" postupcima iz primarnih otopina, u okviru čega je predložio nove koncepte njihovog dobivanja poput *Polymer Complex Method*, *Soft Processing* i *Hydrothermal carbon*. Razvio je originalni pristup formiranju keramičkih presvlaka na metalnim podlogama (GIL), a u zadnje vrijeme bavi se bio-nano materijalima koji obećavaju održivi razvoj društva.

Doktorirao je 1970. godine na *Tokyo Institute of Technology*, a usavršavao se na CNRS laboratorijima: *Odeillo, Orleans* i *Vitry-sur-Seine*, Francuska (1973.-1975.), te na M.I.T.-u, SAD, (1975.-1977.) Godine 1978. izabran je za profesora na Tokyo Institute of Technology (TIT), a za Profesora Emeritusa 2008. godine. Bio je direktor Center for Materials Design na TIT-u (1996.-2003.) Obnašao je mnoge nacionalne i međunarodne dužnosti. Osnovao je i bio na čelu *International Solvothermal and Hydrothermal Association* (ISHA) (2006.-2008.), a trenutno je predsjednik savjetodavnog odbora *The World Academy of Ceramics* (WAC), *Chair Professor* na *National Cheng Kung University*, Taiwan, te *Profesor Emeritus* na *Tokyo Institute of Technology*, Japan.

Nositelj je 30 patenata, a u svojoj znanstvenoj karijeri objavio je preko 700 članaka (od toga 84 revijalna prikaza) u međunarodnim znanstvenim publikacijama, samostalno napisao 3 knjige te je sudjelovao u pisanju 44 knjige. Njegovi radovi citirani su više od 11000 puta, a h-index ima vrijednost 53. Bio je mentor 56 doktoranada (od toga 16 studenata izvan Japana), te više od 70 magistranta.

Prof. Yoshimura primio je brojne nacionalne i međunarodne nagrade među kojima su: *Powder and Powder Metallurgy Society of Japan* (1987. i 1994.), *Richard M. Fulrath Award of the American Ceramic Society* (1987.), *Centennial Anniversary Academic Award of the Ceramic Society of Japan* (1991.), *The International ECERS Award of the European Ceramic Society* (2001.), *Lee Hsun Award of the Institute of Metal Research, Kina* (2008.), *Okazaki Memorial Award of the Fulrath-Okazaki Foundation* (2011.) Profesor Yoshimura član je *The World Academy of Ceramics*, *The American Ceramic Society*, te *Materials Research Society of India*.

Sažetak predavanja: Soft Processing for Sustainable Society – Possibility of Water-Based Processing for Advanced Inorganic Materials

Modern human societies have been developed by the advanced materials and advanced engineering systems both of which consume huge amounts of materials and energy resources. We, however, cannot continue those ways for future sustainable societies because the Earth, the natural system cannot admit further consumption of natural resources and exhausting(wasting) of materials and heats. We, therefore, must search another direction of developments in science and technology.

Advanced ceramic materials have been used in wide area of applications like structural, mechanical, chemical, electrical, electronic, optical, photonic, biological, medical areas. They have generally been fabricated by so-called high-technology, where high temperature, high pressure, vacuum, molecule, atom, ion, plasma, etc. have been used for their fabrications. We have challenged to fabricate those advanced ceramic materials in low energetic ways using aqueous solutions. Since 1989 when we found a method to fabricate BaTiO₃ film on Ti substrate in a Ba(OH)₂ solution by Hydrothermal Electrochemical[HEC] method at low temperatures of 60-200 C, we proposed in 1995 an innovative concept and technology, “Soft Processing” or “Soft Solution Processing,” which aims low energetic (=environmentally benign) fabrication of shaped, sized, located, and oriented ceramic materials in/from solutions. It can be regarded as one of bio-inspired processing, green processing, or eco-processing. When we have activated/stimulated interfacial reactions locally and/or moved the reaction point dynamically, we can get patterned ceramic films directly in solution without any firing, masking or etching. Those Direct Patterning methods differ from previous patterning methods which consist of multi-step processes, for example: (1) synthesis of particles of compounds or precursors, [When this synthesis is done in a solution it is called “Soft Chemistry”, (2) dispersion of the particles into a liquid (“ink”), (3) patterning of the particles on a substrate by printing of the “ink”, (4) consolidation and/or fixing of the particles pattern by heating and/or firing at high temperatures, (5) Those processes would cause cracking and/or peeling of patterned films due to the 3-dimensional shrinkage of printed powders by sintering during heating and/or firing. The notable feature of Direct Patterning is that each reactant reacts directly on site, at the interface with the substrate. Therefore, the chemical driving force of the reaction, $A+B=AB$, can be utilized not only for synthesis but also for crystallization and/or consolidation of the compound AB. It is rather contrasting to general patterning methods where thermal driving force of firing is mostly used for the consolidation of the particles.

We have developed the Direct Patterning of CdS, PbS, and CaWO₄ on papers by ink-jet reaction method and LiCoO₂ by electrochemically activated interfacial reactions. Furthermore, we have succeeded to fabricate BaTiO₃ patterns on Ti by a laser beam scanning and carbon patterns on Si by a needle electrode scanning directly in solutions. Recent success in TiO₂ and CeO₂ patterns by Ink-jet deposition, where nano-particles are nucleated and grown successively on the surface of substrate thus become dense even below 300 C, will be presented. Transparent films of several hundred nm thick can be obtained by 20 times of ink-jet scanning during 15-30 min. As a development of Hydrothermal Electrochemical[HEC] method, we have proposed a new strategy: “Growing Integration Layer[GIL] method”, which can provide well-adhered integrated/graded layers: Titanate/TiO_x/Ti or Titanate/TiO_x/Ti-alloys and/or metallic glass(es) at RT-150 C in a solution. This [GIL] strategy can be applied for many areas of functional ceramics. In addition, our recent results on size and shape controlled mono-dispersed nano-particles of CeO₂, (Hf,Eu)₂O_{3-x}, Fe₃O₄, etc. will be presented. In those processes, the importance of “Complex Formation” by chelating agents will be clarified in the comparison of conventional “Sol-Gel” methods with “Polymerizable Complex” methods, which we proposed in 1992 and later.

Sažetak predavanja: Growing Integration Layer [GIL] Strategy: Direct Fabrication of Compositionally, Structurally and Functionally Graded Ceramic Coatings and/or Films from Mother Materials in Solution

We have proposed a novel concept and technology of the formation “Growing Integration Layer” [GIL] between ceramics and metallic materials to improve the adhesion performance. Those GIL(s) can be prepared via integration of ceramic film formation from a component of the metallic materials by chemical and /or electrochemical reactions in a solution at low temperature of RT-200°C. They have particular features: 1) Widely diffused interface(s), 2) Continuously graded layers grown from the bulk(substrate), 3) Low temperature process, etc. They are quite different from Layer-by-Layer[LBL] strategy, where every layer is deposited from the Top. BaTiO₃ or SrTiO₃/TiO_x GIL films on Ti plates formed by hydrothermal-electrochemical method showed good adhesion. CaTiO₃/Al₂O₃/Ti₂Al GIL films on TiAl exhibited excellent adhesion and anti-oxidation performances: they could be sustained for 10 times cyclic oxidation test at 900 C in air for 5 hrs. The GIL strategy is effective for many metallic alloys and bulk metallic glasses because they generally contain active component(s). On a Ti-base Bulk Metallic Glass, we could succeed to prepare bioactive titanate nano-mesh layer by hydrothermal-electrochemical techniques at 90-120°C. Similarly, bioactive oxide layers could be prepared on different Bulk Metallic Glasses. Carbon film formation on SiC can be regarded as one of GIL methods.

The GIL methods are typical “Soft Process” and “Green Process” using aqueous solutions, and applicable for various functional and structural ceramics layers.