別紙４（課程博士）

Date of Submission:
平成 28 年 08 月 28 日

Department 機械工学専攻	Student ID Number 学籍番号	第 139109 号
Applicant's name 氏名	TOIBAH BINTI ABD. RAHIM	

Supervisors 指導教員	Masahiro Fukumoto Toshiaki Yasui

Abstract

論文内容の要旨 （博士）

Title of Thesis 博士学位論文名	Fabrication of Cold Spray Ti-O Coatings Engineered from Agglomerated Powders （凝集粉末作製技術によるコールドスプレーTi-O 皮膜の創製）

(Approx. 800 words)
(要旨 1,200 字程度)

Current attention has focused on the preparation of thick ceramic coating using nanostructured materials as feedstock materials using thermal spray process. Cold spray method has appeared as a promising process to form ceramic nanostructured coating without significantly changing the microstructure of the initial feedstock materials whereas many conventional thermal spray processes do due to its low processing temperature. However, deposition of ceramic powders by cold spray is not easy due to brittle characteristics of the material. Moreover, the bonding mechanism on how the ceramic coating was formed on the substrate is still unclear as this method requires plastic deformation of particles upon the impact onto the substrate.

Therefore, in this study, focused have been made on the TiO_2 nanostructured feedstock materials which were synthesized throughout of the study. The properties of the powders also have been altered by several conditions in order to make it suitable for cold spray deposition. The mechanism of coating deposition and properties of the feedstock powders were investigated in this study. The following results which obtained by this study were summarized as below:

1. In this work, the synthesis of agglomerated TiO_2 powders, which are ready to be used as feedstock materials for a cold spray process after synthesis via a simple hydrolysis (TiO_2-H) and hydrothermal (TiO_2-HT) process, is described. The XRD patterns showed that single phase anatase TiO_2 was able to be produced using a low temperature process for the hydrolysis and hydrothermal methods. However, the results showed that TiO_2-H powders have a smaller crystallite size and broader peaks compared with TiO_2-HT powders. SEM and TEM analysis confirmed that the TiO_2-H powders were built up from nano-sized particles, and were further agglomerated into micrometer-size, which is a preferable size for the cold spray process. On the other hand, TiO_2-HT powders showed a formation of agglomerated particles with minimal particle agglomeration which was revealed by the SEM image and the particle size analyzer. A preliminary study on coating deposition using cold spray showed that TiO_2-H powders can be deposited onto ceramic tile substrate with a ~50μm thickness. Meanwhile for TiO_2-HT powders, only particle embedment can be observed on the surface of the substrate. The results reveal that porosity contained in the agglomerated morphology is important in order to build up the coating by cold spray due to the tendency of the porous structure to break easily upon impact onto the substrate.

2. To further clarify the effect of porosity contain in the powder for the cold spray deposition, effect of low calcination on the as-synthesized TiO_2 by hydrolysis method have been conducted. Then, as-synthesized TiO_2 and calcined TiO_2 powders were studied on coating deposition by cold spray process. The results of this study indicated that a post treatment on TiO_2 powder improved powder deposition on ceramic tile substrate via the cold spray method. The cross-section of the obtained coating which was observed using SEM showed that nanoparticles TiO_2 powders in the agglomerated form were able to be deposited on the substrate and formed a thick coating. A stacking of agglomerated TiO_2 powders was found on the cross-section observation which is due to the breaking up of ceramic particles which was induced by porosity in the powder and is believed to be responsible for the formation of the coating. The results of this study also reveal that, when the feedstock powders have denser packing of particles and minimum number of porosity in the powder, breaking of particles during the spraying become more difficult. This hard and dense particle made them resistant to fragmentation and adherence on surface of the substrate.
3. Further study have been conducted by addition of ammonium sulfate; $(NH_4)_2SO_4$ during the powder synthesis. Addition of structure-directing agent, $(NH_4)_2SO_4$ promotes the agglomeration to occur with denser closed packing of particle arrangement which reduce the number of existing porosity in the synthesized powder. The addition of $(NH_4)_2SO_4$ addition was found to be very effective to unite the nano-sized particles together to form agglomeration in order to form the tertiary particles. The preliminary study of coating formation depicted that the powder obtained could be used as the feedstock powder for cold spray process to make coating as it can be deposited onto the ceramic tile substrate. Once again, porosity in the powders was deduced as one of the crucial factors that contribute to better deposition of TiO_2 coating by cold spray process. Plastic deformation also may contribute to the formation of coating due to the used of nanostructured powders which received high local compact pressure during the spraying process.
4. Further studies on the obtained coating have been investigated. The study reveals that the properties of the coating (hardness, roughness and porosity) also depend on the properties of the initial feedstock powders. Moreover, anatase phase was preserved as revealed by the XRD analysis. This finding proves that cold spray process is suitable process to fabricate TiO_2 coating which can prevent phase transformation to occur due to low processing temperature. Details observation on the surface and cross-section of the coatings show that nanostructured particles from the feedstock powders were well-retained in the coating structure.
5. In order to study the individual particle impact morphologies, wipe tests were conducted on aluminum, copper and ceramic tile substrate. From the SEM observation, the results showed that the collided particles were plastically deformed and adhered on the hard ceramic tile substrate during deposition. However, in the case of aluminum and copper substrate, the splat diameters were smaller than the feedstock powder size and both particles and substrates were deformed during the collision. Moreover, many craters were observed on these metal substrates. It was found that the deposition behavior of TiO_2 particle and the crater formation by the cold spray process was affected by the hardness and surface roughness of the substrate materials.