別紙４（課程博士）

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Department 機械工学専攻	Student ID Number 学籍番号	第 093255 号
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Abstract

論文内容の要旨 (博士)

Title of Thesis 博士学位論文名	固相粒子積層法により作製した金属皮膜の特性評価と粒子堆積機構 （Evaluation of metallic coating by solid particle deposition process and mechanism of particle deposition）

(Approx. 800 words)

(要旨 1,200 字程度)

コールドスプレー(Cold Spray: CS)法とは特殊ノズルによって超音速に加速された作動ガス流に材料粉末を投入・加速し，粒子を基材に衝突・堆積させることにより皮膜を作製する成膜法である．作動ガスの温度を材料粉末の融点以下に抑え成膜することから，材料粉末の酸化や熱変質を抑制でき，材料粉末の特性を損なわずに皮膜を形成することが可能である．また，密着強度が高く成膜速度に優れることから厚さ数cm単位の高品位な皮膜を作製でき，単なる成膜技術としてだけではなくバルク体創製技術や補修技術としても期待されている．現在，国内外の研究機関において盛んに研究が行われているが，実用例は少なく，工業的な信頼を得るためにさらに詳細なメカニズムや現象の解明が求められている．そこで本研究ではCS法の粒子堆積機構の解明を目的として，成膜の素過程である単一粒子の付着メカニズムから複数粒子が積層し厚膜を形成する成長過程までの各過程に関しての粒子の積層機構を検討し，皮膜特性に与える影響を評価した．

本研究で得られた知見を以下に示す．

1. 粒子間結合機構の解明を目的とし，Al- Cu複合皮膜を作製し，異種金属粒子間界面を観察し，CS法と同様に固相粒子積層法であるエアロゾルデポジション法と比較した．観察結果より，両プロセスの皮膜の異種金属粒子間において金属拡散の兆候が見られたが，エアロゾルデポジション法において金属拡散の兆候が見られた界面は少なく，粒子表面の持つ酸化物層を有する異種金属粒子界面が多く存在した．

2. 数値解析を用いてCS法における重要なパラメータである基材近傍での飛行粒子挙動調査を行った．飛行粒子挙動と成膜現象に関して調査した結果，一定のノズル－基材間距離までガス流の加速を受け，基材近傍での飛行粒子速度は上昇するが，皮膜密着強度は粒子速度の変化に呼応せずに，粒子衝突範囲が狭く積極的な後続粒子の衝突が生じる短いノズル-基材間距離で最大値を示した．

3. 皮膜密着強度と基材加熱により制御した基材温度の関係を皮膜および基材の材料物性や基材表面酸化について評価した結果，基材加熱は密着強度を向上させるためには基材加熱によって成長した基材表面酸化皮膜を粒子の衝突によって除去することが重要であることが確認された．

4. 成膜後の熱処理が皮膜の機械的特性に与える影響を調査した．粒子-粒子間の密着性に着目し，これを皮膜の曲げ強さによって評価した結果，試験片作製と熱処理を行う順序によって曲げ強さが大きく変化した．皮膜-基材間に生じる内部応力が皮膜特性を大きく左右することが示唆された．

5. コールドスプレー皮膜の内部応力をX線回折および基材の変形によって評価した結果，内部応力は皮膜の厚さ方向に応力分布が存在し，特に皮膜-基材界面近傍における応力の不均衡が皮膜密着強度を低下させていることが明らかとなった．

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The cold spray process was developed more than two decades ago and this advanced coating process has been rapidly developed. This process enables to produce thick and high purity metallic coatings under atmospheric ambient with extremely high deposition rates. Conventionally, a thermal spray process has been used to produce thick coatings in a wide range of industrial applications. However, the thermal spray coatings are formed through the deposition of molten and semi-molten feedstock powder materials. The melting metallic material is easily oxidized or azotized under atmospheric ambient, then the spray materials lose original properties. The solid particle deposition processes: the cold spray process and the aerosol deposition process overcome this problem by avoiding material melting. The aerosol deposition process uses mainly a ceramic fine powder as the feedstock powder material and the powder material deposits in a vacuum condition. The feedstock powder materials deposit on the substrate without heating. On the other hand, the cold spray process uses mainly a soft metal powder as the feedstock powder materials and the powder materials deposit in an atmospheric condition. This process uses electrically heated supersonic gas (nitrogen, helium or air) stream with lower temperatures than the melting point of feedstock material. The feedstock powder materials are accelerated by the gas stream through a De-Laval nozzle towards a substrate. The feedstock powder materials collide and deposit on the substrate surface in the solid phase. Currently, this process has been made actively studied in research institutions in the world. However, the practical applications are few. Thus, elucidation of more detailed mechanism and phenomena in order to obtain industrial reliability are required. For example, because of the solid state particle deposition, the electrical and chemical properties of the cold-sprayed coatings are almost similar to the bulk materials. On the other hand, the mechanical properties are different from the bulk materials due to the severe plastic deformation of the particles. The residual stress and the bonding state of the particles in the coatings can be considered to be additional reason for the coating property. However, the formation mechanism of residual stress is not entirely clear. Therefore in this study, the elucidation of particle deposition mechanism of the cold spray process was selected as a main purpose.

The influence of spraying behavior on characteristics of coating was investigated throughout of the deposition process: from single particle bonding to building up of coating.

The results obtained in this study are summarized as below:

(1) To investigate the mechanism of single particle bonding, a cold sprayed Cu-Al composite coating was compared microscopically with a coating sprayed by the aerosol deposition process. The aerosol deposition process is solid particle deposition process without material heating. Metal diffusion has been observed between dissimilar metal particles of the coating by both deposition processes. However, the aerosol deposited coating generally showed bonding mediated by oxide layer of particles. The aerosol deposition process generates a small amount of newly-formed surface.

(2) The gas flow and in-flight particle behaviors were analyzed by the computational fluid dynamics. The analysis results showed that a particle velocity is increase by increasing standoff distance until specific distance. However, coating adhesion strength decrease by increasing standoff distance. The shorter standoff distance showed higher adhesion strength. As reason of this, increasing collision of subsequent particle and increasing heat input by gas stream were suggested.

(3) To investigate influence of temperature at bonding on adhesion of coating, the substrate temperature was controlled by substrate heating during spraying. Cu, Al and Fe particles sprayed on heated Al alloy, Cu and Fe substrates. The substrate heating showed potential to enhance coating adhesion. However, it was limited for material combinations: Cu particle-Al alloy substrate, Cu particle-Fe substrate, Fe particle-Cu substrate and Al particle-Al alloy substrate.

(4) A heat treatment after the coating formation was investigated for the effect on the mechanical properties of the coating. To focus bonding mechanism between particle and particle, the bending strength of coating was examined. As result, the bonding strength was significantly changed by sequence of bending specimen preparation and heat treatment. As reason of this, a distribution of internal stress of coating was suggested.

(5) The distribution of internal stress of the coating was examined by X-ray diffraction and substrate deformation. As a result, the internal stress is non-uniform in the direction of coating thickness. Especially, there is disproportion of internal stress at the coating interface. It was suggested as a main factor for decreasing the adhesion strength.