Chapter

Influence of Annealed Aluminum Properties on Adhesion Bonding of Cold Sprayed Titanium Dioxide Coating

Noor Irinah Omar, Motohiro Yamada, Toshiaki Yasui and Masahiro Fukumoto

Abstract

It is well known that cold spraying ceramic materials can be difficult because cold spraying requires plastic deformation of the feedstock particles for adhesion to the substrate. The challenge lies in the difficulty of plastically deforming hard and brittle ceramic materials, such as TiO_2. Previous studies have reported the possibility of cold spraying thick pure TiO_2 but the bonding mechanism of cold sprayed TiO_2 is not fully understood. The factor like substrate condition as oxide film thickness and mechanical properties may also affect cold spray deposition but not fully understood in cold spraying ceramic. The aim of the present research is to investigate the correlation between the oxide thickness and substrate deformation with the adhesion strength of cold-sprayed TiO_2 coatings toward the bonding mechanism involved. Relevant experiments were executed using Al 1050, subjected to various annealing temperatures and cold-sprayed with TiO_2 powder. The results indicate a decreasing trend of coating adhesion strength with increasing annealed substrate temperature from room temperature to 400°C annealed. Metallurgical bonding is pronounced as bonding mechanism involved between TiO_2 particle and annealed 1050 substrate.

Keywords: cold spray, bonding mechanism, pure aluminum, titanium dioxide, annealed substrate, metallurgical bonding

1. Introduction

Cold spraying, also referred to as kinetic spraying, represents a relatively novel technique for material deposition that has been in development for over twenty years. The process involves the high-velocity acceleration of powder particles, typically in the 300–1200 m/s range, in a jet flow of supersonic velocity with the projection directed onto a substrate or a coating that has been pre-deposited at an absolutely solid state. Due to the impact at high velocity, intensive deformation of plastic manifests in the cold-sprayed particles or substrate, allowing the formation of a low oxidized cold-sprayed coating [1]. Since nanostructured TiO_2-anatase coatings have an extensive active surface and chemical stability, and are at a comparatively reasonable cost, they are utilized as functional material [2]. The crystalline framework of TiO_2 has a major impact upon its photocatalytic performance where TiO_2 in anatase stage supplies greater photocatalytic activity than in its rutile stage. At temperatures exceeding 900°C, which is above the melting point of TiO_2 (1908°C), the anatase stage irreversibly changes into the rutile stage. At temperatures above the melting point of TiO_2, the deposition of molten or semi-molten droplets form thermal impossible to avert the phase transformation of TiO_2 in thermal spray procedures [2]. Therefore, cold spraying is the best solution because it sprays below the material melting temperature.

In terms of the mechanism for bonding, numerous works have been conducted in the past 10 years to enhance our understanding of this phenomenon [3–14]. At present, the manifestation at the interface of adiabatic shear instability, (ASI) represents the most accepted bonding-mechanism theory, resulting from the high rate of strain as well as the intense local deforming that occurs through the process of depositing the particles. In the vicinity of adiabatic shear instability occurrence, adiabatic heating-induced thermal softening dominates the hardening through work, and therefore the metals' behavior is similar to viscous material and leads to extrusion from the interface, with the formation of an outward metal jet at the rim [4–5]. The presence of the metal jet with viscosity assists in the cleaning of the native-oxide film that is cracked and originates from the surfaces of particle or substrate, facilitating contact between the metals and therefore the occurrence of metallic bonding [6]. Two mechanisms widely perceived to dominate in terms of metallic bonding in cold spray are interlocking through mechanical means and bonding metallurgically.

It is interesting that cold spraying can also be used to deposited ceramic materials, although initially it appears impossible as cold spraying require plastic deformation to work. Previous studies have reported the possibility of cold spraying thick pure titanium dioxide, TiO_2 within range 300 μm [15] but the bonding mechanism of cold sprayed TiO_2 is not fully understood. Several experiment result regarding the bonding mechanism of cold spraying TiO_2 particles onto metal and ceramic substrates have been published. Toibah et.al reported as-synthesized TiO_2 powders that calcined at 200°C and 300°C showed the successful deposition of TiO_2 coating on the ceramic tile substrate by the cold spray method. The coating deposition occurred due to tamping effect through slipping of nanoparticles due to high impact during the particle collision. Mechanism responsible for the TiO_2 deposition is chemical bonding between TiO_2 particles and substrate or among particles during cold sprayed process may lead to coating formation [16]. Kliemann et al. used 3-50 μm TiO_2 agglomerates formed from 5 to 15 nm primary particles for the continuous coating on pure titanium, stainless steel, copper and aluminum alloy substrate. They identified ductile substrate that allow shear instability to happen as primary bonding mechanism between the particles and the substrate [17]. Gardon et al. claimed that the mechanism responsible for the TiO_2 deposition by the cold spraying process on the stainless-steel substrate is the chemical bonding between the particles and the substrate. They showed that the previous layer of titanium sub-oxide prepares the substrate with the appropriate surface roughness needed for the TiO_2 particle deposition. Moreover, the substrate composition is also important for the deposition because it can provide chemical affinities during the particle interaction after impact. The substrate hardness may also ease the interaction between the particles and the substrate [2].

Based on the above mention meaningful findings, it shown a lot of factor influenced toward bonding mechanism of cold sprayed TiO_2 coating and one of it is substrate surface. Previous studies reported the possibility of cold spraying thick pure TiO_2 but the bonding mechanism of cold sprayed TiO_2 is not fully understood. The factor like substrate condition as oxide film thickness and mechanical properties may also affect cold spray deposition but not fully understood in cold spraying ceramic. Therefore, this study investigated the effect of annealed substrate properties Al 1050 toward bonding mechanism of cold sprayed TiO_2.

2. Materials and methods

2.1 Process

In all coating experiments, cold-spraying equipment with a De-Laval 24TC nozzle (CGT KINETIKS 4000; Cold Gas Technology, Ampfing, Germany) was used. Nitrogen was used as the process gas with a 500°C operating temperature, and a 3 MPa pressure. The spray distance was 20 mm, with a process traverse speed of 10 mm/s. The coatings were deposited on grit-blasted annealed pure aluminum (Al 1050). The substrates were annealed with an electric furnace to preheat the grit-blasted substrate to four different temperatures respectively (i.e., 100°C, 200°C, 300°C and 400°C) before spraying. In all cases, the temperature of the annealed substrate during spraying was room temperature.

2.2 Materials

As a feedstock, we applied agglomerated TiO_2 powder (TAYCA Corporation, WP0097) containing a pure anatase crystalline structure with an average particle size of about 7.55 μm. The material chemical composition of substrates used is presented in **Table 1**.

2.3 Characterization

2.3.1 Tensile-strength testing

In accordance with JIS H 8402, specimens measuring Ø25 mm × 10 mm were used to assess the coatings' adhesion strength, given as the fracture load value measured by a universal testing machine (Autograph AGS-J Series 10 kN, Shimadzu). We measured the adhesion strength over an average of five specimens for each of the spraying conditions.

2.3.2 Coatings evaluation

A scanning electron microscope (SEM: JSM-6390, JEOL, Tokyo, Japan) was used to observe the TiO_2 coating's cross-sectional microstructures on annealed substrates. The observation sample of the TiO_2 coating was prepared by embedding a 25mmx10mm sample into a hardenable resin. The hardened sample embedded in hardened resin was ground with silica papers to a #3000 grit size and finally polished with 1 μm and 0.3 μm alumina suspension.

	Al	Fe	Si	Cu	Mn	Mg	Zn	Ti	Other total
Al 1050	Bal	0.40	0.25	0.05	0.05	0.05	0.05	0.05	0.03

Table 1.
Material chemical composition (wt.%).

Measured regions	Al 2p,	O1s,
Measured X-Ray output [W]	10	
Probe diameter[μm]	50	
Time per step [ms]	30	
Pass energy	140	
Cycle	30	

Table 2.
XPS parameters for substrate oxide layer analysis.

2.3.3 Micro-Vickers hardness

To investigate the relationship between the annealed substrate surface hardness and the adhesion strength of the TiO_2 coating on the annealed substrate, the substrate hardness was measured using an HMV-G micro-Vickers hardness tester (Shimadzu). The measurement showed a hardness of HV 0.1; the test load on the cross section was 98.07[mN]. The final micro-hardness value was the average of 5 tests taken at approximately the same points for each substrate.

2.3.4 Substrate oxide evaluation

X-ray photoelectron spectroscopy (XPS) is a versatile surface analysis technique used for compositional and chemical state analyses. In this study, XPS analysis (ULVAC-PHI, PHI Quantera SXM-CI) using a monochromatic Al Kα source (15 mA, 10 kV) was performed. Wide (0–1000 eV) and narrow scans of Al 2P and O1s for different annealed substrates were collected. The measured binding energies were then corrected with C 1 s at 285.0 eV. When pre-sputtering to clean the surface was performed, the sample surface was reduced and the measurements were affected, so XPS analysis was performed without pre-sputtering. **Table 2** shows the XPS analysis conditions for substrates oxide analysis.

2.3.5 Wipe test

A CGT Kinetiks 4000 cold-spray system (Cold Gas Technology, Ampfing, Germany) with a custom-made suction nozzle was used to perform the wipe test and coating using TiO_2 powder onto the annealed 400°C pure aluminum substrates. The wipe test was conducted to study the deformation behavior of a single particle on this substrate. Prior to deposition, substrate was ground and polished until a mirror finish surface was obtained. The process gas temperature and pressure used were 500°C and 3 MPa, respectively. Nitrogen was used as the process gas. The distance between the exit of the nozzle and the substrate was fixed at 20 mm. The traverse speed of the process was 2000 mm/s. Prior to spraying, the substrates were rinsed with acetone. An FEI Helios Dual Beam 650 field emission SEM (FESEM) and focused ion beam (FIB) microscope was used to investigate the single particle TiO_2 deposition onto mirror polish annealed substrate.

3. Results and discussion

3.1 Strength of adhesion

The adhesion strength of the cold-sprayed TiO_2 coating on annealed Al 1050 pure aluminum are shown in **Figure 1**. The TiO_2 coating on the annealed soft substrates showed a decreased trend of adhesion strength from room temperature to 400°C, with values from 3.88 to 2.05 MPa. The increase of annealed substrate temperature also showed a thicker surface oxide on substrate surface.

3.2 SEM cross-section microstructure of TiO_2 coatings on annealed Al 1050 substrates

Figure 2a–e shows the TiO_2 coating cross-sectional area on Al 1050 for various substrate annealing temperatures. Under all condition figures, the figures show a dense coating with a thickness in the range of 200 to 300 μm, indicating that a critical velocity was reached for this soft material. This suggests that the TiO_2 coating adhered well to the annealed Al 1050 substrate from room temperature to 400°C annealing.

We can categorize the cold-spraying procedure into two stages: (1) adhesion and (2) cohesion bonding. Adhesion or the formation of the interface between the substrate and particle is the first stage. The annealed substrates can clearly implement this stage, which forms the first coating layer, particularly for the soft material, Al 1050.

3.3 Substrate Vickers microhardness

Figure 3 shows the annealing substrate hardness of Al 1050 from room temperature to 400°C. The pure aluminum, Al 1050 showed a decreasing trend from 45.36Hv for room temperature to 27.70Hv for 400°C annealing.

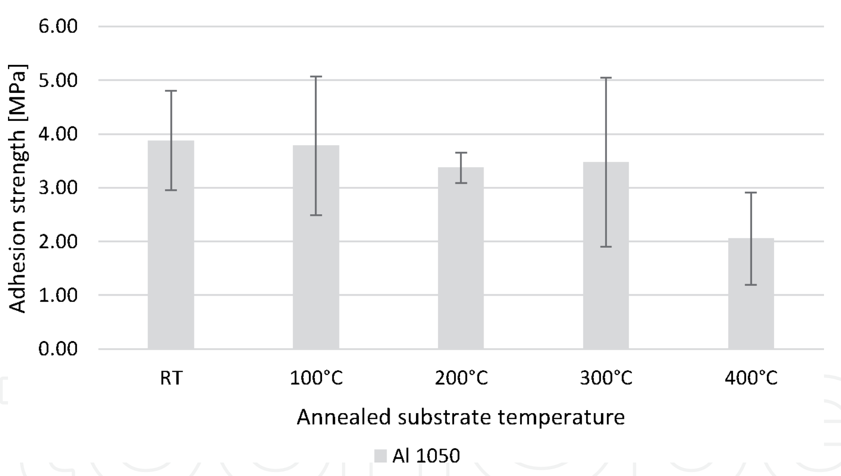

Figure 1.
Adhesion strength of the TiO_2 coating on annealed Al 1050.

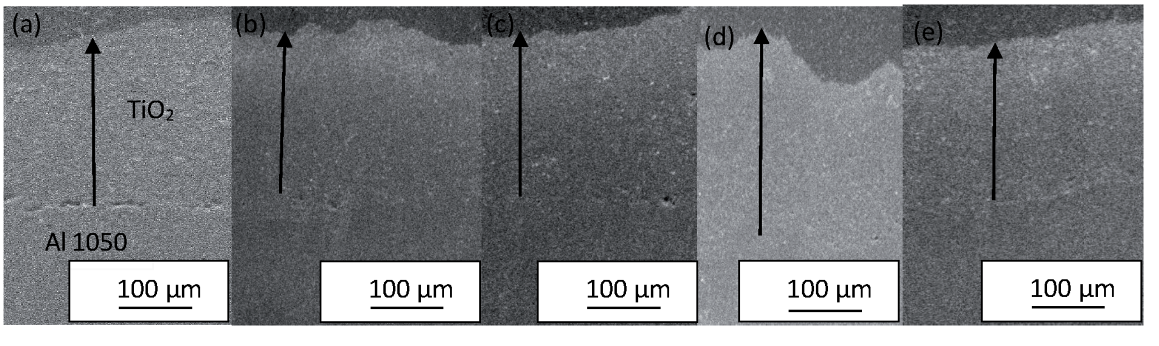

Figure 2.
Cross-section microstructure of TiO_2 coatings on Al 1050. (a) Room temperature; (b) annealed 100°C; (c) annealed 200°C; (d) annealed 300°C and (e) annealed 400°C.

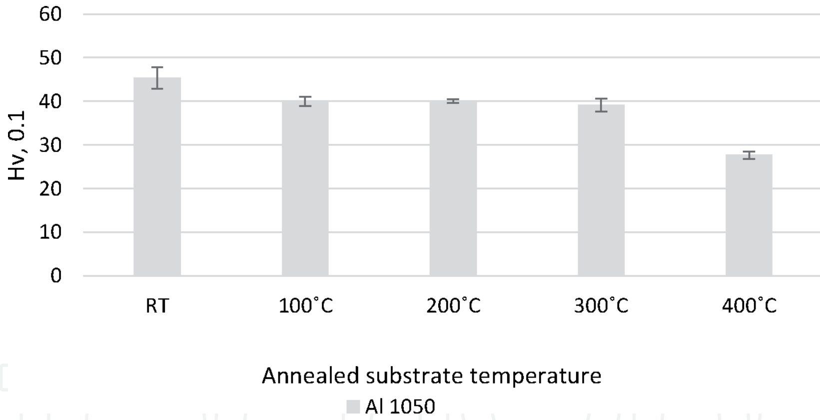

Figure 3.
Annealed substrate microhardness of Al 1050 from room temperature to 400°C- annealed.

This trend appears because pure materials experienced recrystallization process at 400°C and slow cooling in the electric furnace; allow the grains to growth larger and reduce the substrate hardness. Based on decrease trend shown by adhesion strength TiO_2 coating on annealed pure aluminum, substrate deformation (mechanical anchoring) due to TiO_2 particle impacting during cold spraying is not the main factors that influence adhesion bonding between TiO_2 and annealed pure aluminum.

3.4 Depth profile of the oxide layer on Al 1050 substrate

The results of the depth analysis of room temperature substrate and annealed 400°C by X-ray photoelectron spectroscopy for Al 1050 substrates is shown accordingly in **Figure 4**. The composition as a function of depth can be analyzed by in-situ argon ion beam sputtering, found on most surface analytical equipment.

Al 1050 as shown by **Figure 4(a)** and **(b)**, shown that the atomic composition of Oxygen in the deepest part of the oxide layer increases as the annealing substrate temperature increases from RT to 400°C. This indicate that the oxide layer of pure aluminum grows thicker as the annealing substrate temperature is increased. According to W. Ya Li et.al stated that the increasing of oxide film thickness, it will need more kinetic energy to break up and extrude the oxide film, thus a higher particle velocity is needed for bonding. In other words, the effective bonding area is decreased under the same particle impact conditions [18]. It could explain the decreased trend of adhesion strength TiO_2 coating on 400°C annealed pure aluminum because the particle velocity was constant in all condition in this experiment.

3.5 Oxide layer composition

X-Ray photoelectron spectroscopy (XPS) is a versatile surface analysis technique that can be used for compositional and chemical state analysis. In this experiment, total of 30 cycles is involved and to plot binding energy graph versus intensity to study the chemical state changes, outermost surface referring to the 1st cycle data, mid-layer referring to the 15th cycle data and deepest part referring to the 30th cycle data for each substrate conditions.

The peak position of the aluminum oxide layer, Al_2O_3 on outermost surface at 74.8 eV and aluminum metal at approximately 72.8 eV was prominently present in the mid-layer and deepest part for the room-temperature substrate Al 1050 shown by **Figure 5(a)**. The peak position of the O1s shown by **Figure 5(b)** also support the present of aluminum oxide layer, Al_2O_3 on outermost surface at 531.3 eV and

Figure 4.
Depth profile analysis of Al 1050 (a) room temperature (b) 400°C annealed.

slightly presented of aluminum in hydroxide on mid-layer and deepest part given by 532.4 eV, supported that substrate mostly Al metal state for mid layer and deepest part at room temperature substrate. Meanwhile, at the annealing temperature of 400°C for Al 1050 substrate, **Figure 6(a)**, the peak position of aluminum oxide layer, which was at approximately 74.8 eV, was detected at the outermost surface, mid-layer, and deepest part of the substrates. O1s supported that by the present of aluminum in hydroxide/oxyhydroxide state at 532.4 eV [19]. This indicate Al 1050 substrate experienced chemical composition changes from Al metal state in room temperature substrate to aluminum in hydroxide condition in 400°C annealed.

3.6 FIB splat TiO_2 particle on 400°C annealed Al 1050

Figure 7 shows the top view of TiO_2 particles on 400°C-annealed Al 1050. This wipe test was conducted to further understand the bonding mechanism of TiO_2 particle on annealed Al 1050 substrate. Only 400°C-annealed Al 1050 was selected because it had the highest oxide thickness. The results obtained revealed that the TiO_2 particle were found unchanged after the collision and the substrate surface of the 400°C-annealed Al 1050 experienced a deformation due to impacting during the cold-spraying process, as shown by **Figure 7**. Soft substrates such as aluminum, the TiO_2 particles were impacting the surface with minimal deformation, and the particles rebounded after the impact, leaving craters on the surface of the substrate. This shown by arrow in the **Figure 7**. K.-R. Ernst et al. [20] also mentioned that for soft substrate, impact energy that was generated during the spraying process was also used for deformation of the substrate. Since 400°C

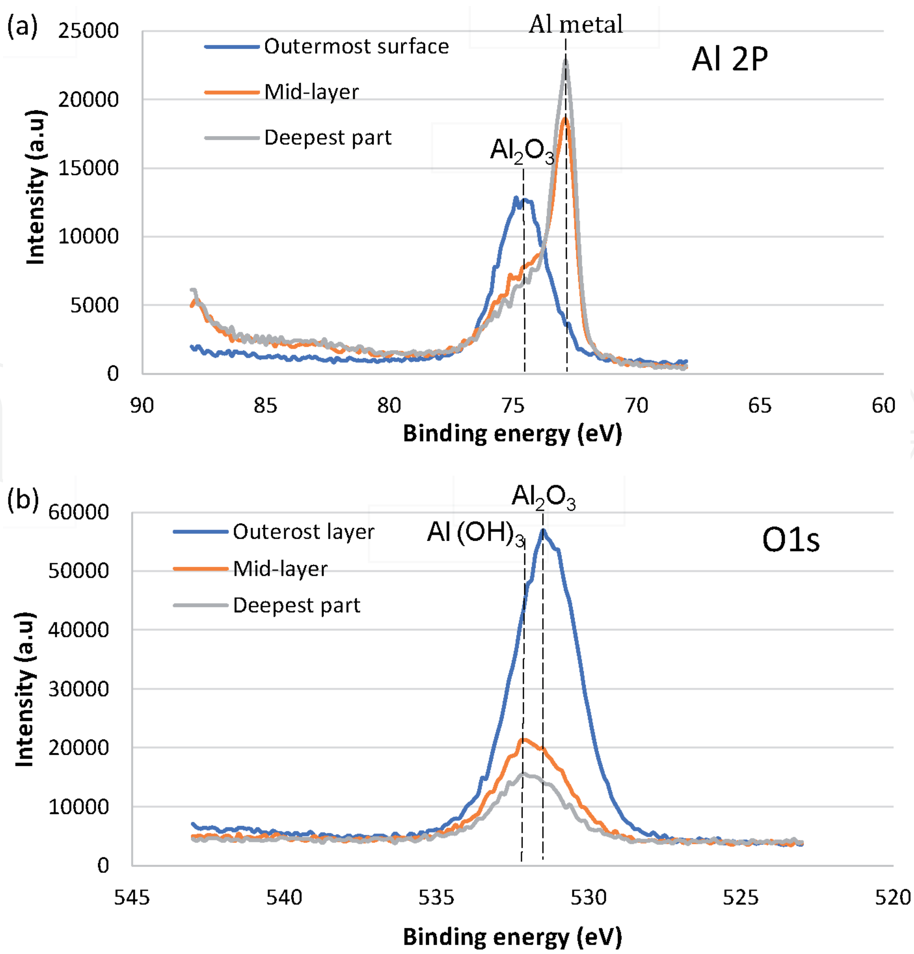

Figure 5.
XPS spectra of Al 2P for (a) room temperature and O1s for (b) room temperature.

annealed aluminum has lower hardness due to recrystallization, the depth of the craters was deeper and experienced heavy damage, as seen in the SEM image in **Figure** 7.

Figure 8 shown a cross-section of a single particle of TiO_2 on 400°C annealed 1050. Referring to **Figure 8**, since the shear instability starts at a position away from the bottom center of a TiO_2 particle, the bottom region of such a deposited particle can be divided into three regions along the particle - substrate boundary: (i) the particle jetted out region,(J) generated by the severe shear plastic strain induced by adiabatic shear instability (ASI); (ii) the well-bonded region (B); where the particle and the substrate are intimately bonded and (iii) the rebound region (R) where the shear instability did not occur and the accumulated elastic energy from the impact of a sprayed particle detached the particle from the substrate. At the boundary of B and R, ASI is accompanied by severe shear stress, and an abnormal increases in temperature can easily expel the particles, and consequently the oxide covering the surface of particle or substrate can be broken and removed [3, 4, 11, 21–24].

The adhesion strength of the TiO_2 coating on annealed Al 1050, showed a decreased trend as the annealed substrate temperature is increased from room temperature to 400°C annealed. This indicate that substrate deformation or mechanical anchoring is not one of factors that influence the adhesion bonding for annealed Al 1050 with TiO_2 at an annealing temperature of 400°C. Referring to arrow indicate in **Figure 8**, there were a gap observe between deposited TiO_2 particle and 400°C annealed 1050 substrate at rebound, R and bonded, B region. This result indicate that even severe plastic deformation is occurred due to TiO_2 impacting during cold spraying, thin amorphous oxide will remain between particle-substrate [25].

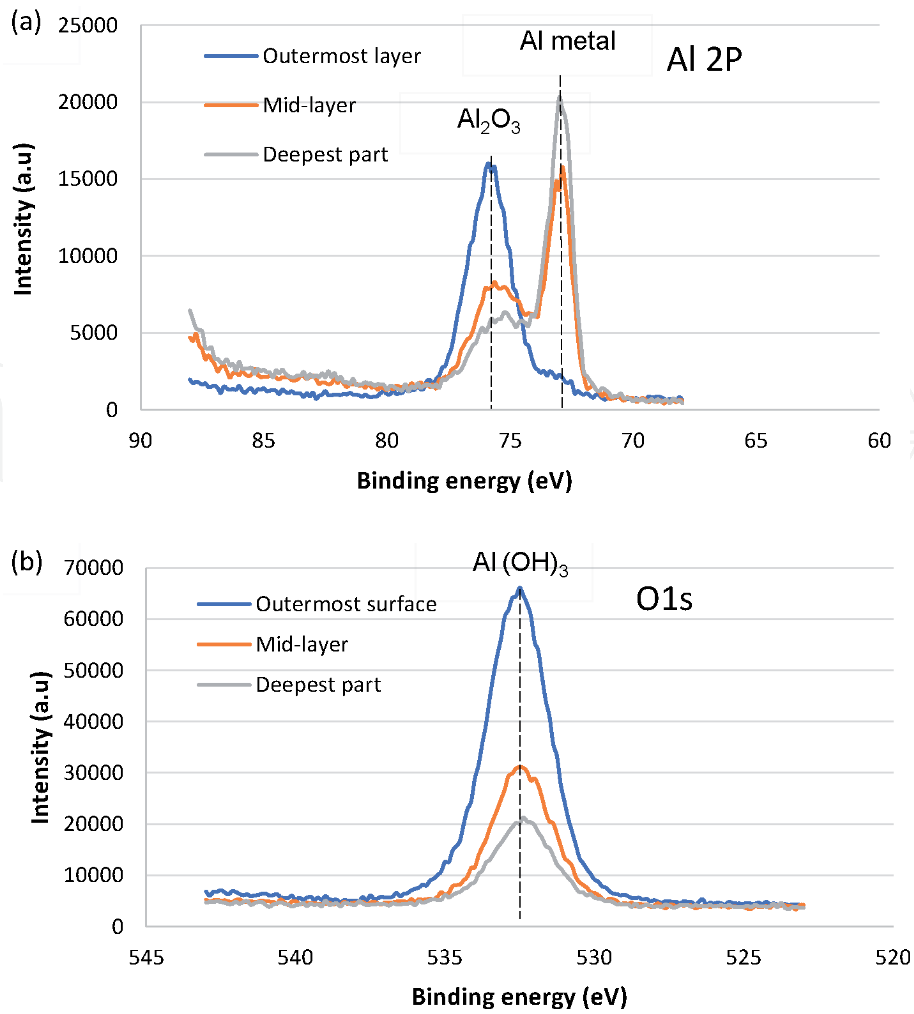

Figure 6.
XPS spectra of Al 2P for (a) 400°C annealed and O1s for (b) 400°C annealed.

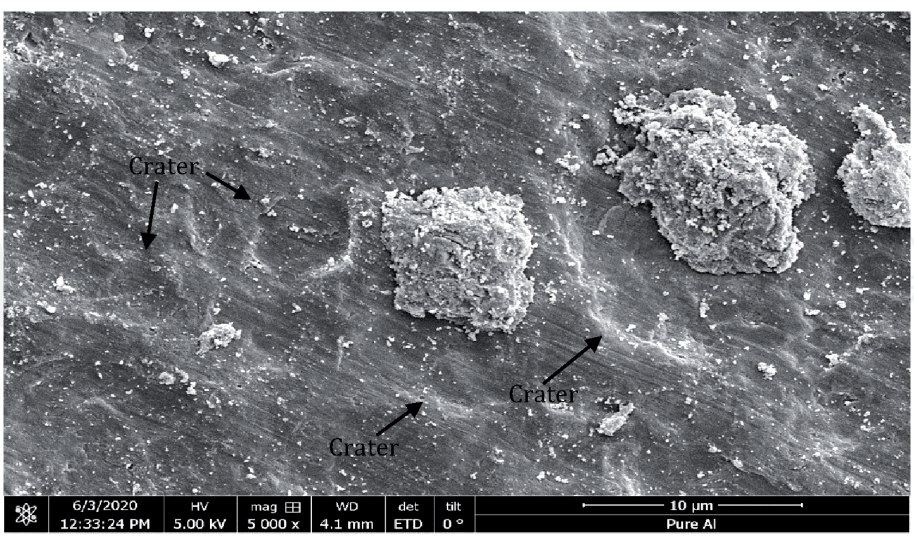

Figure 7.
Top view of TiO_2 particles on 400C annealed Al 1050.

Moreover, due to thicker oxide on 400°C annealed Al 1050 or the fresh surfaces of metallic such as aluminum may be able to be re-oxidized due to their high reactivity with oxygen in air during deformation [25], it prevent bonding to form between deposited TiO_2 particle and deform substrate surface of 400°C annealed Al 1050. This may explain that bonding mechanism involved between TiO_2 coating and

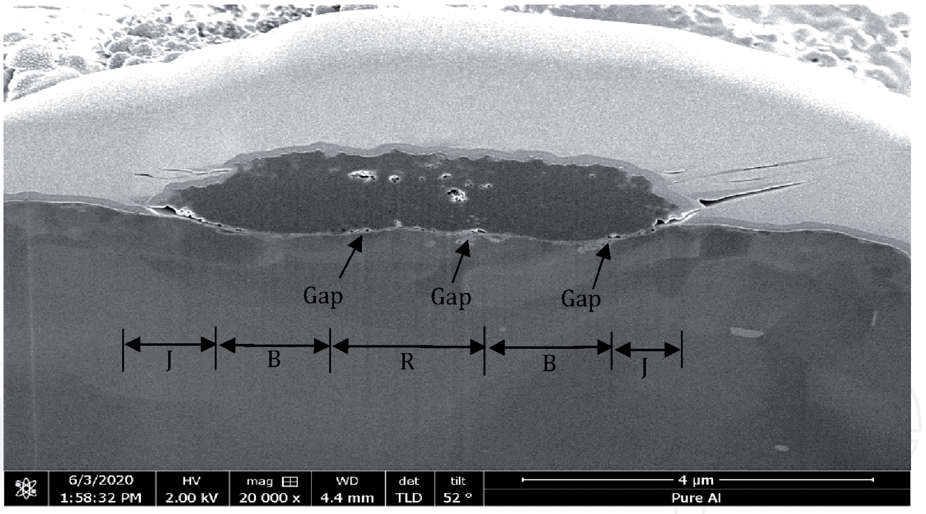

Figure 8.
FIB cross-section of a single particle of TiO_2 on 400°C-annealed Al 1050. J indicates the jetted-out region; B is the bonded region; R is the rebound region.

400°C annealed 1050 is newly formed substrate surface that free from oxide with TiO_2 particle which is similar with the cold spraying of metallic materials that known as metallurgical bonding.

4. Conclusions

This study investigated the correlation between the adhesion strength of cold-sprayed TiO_2 on Al 1050 pure aluminum—annealed at temperatures ranging from room temperature to 400°C.

1. The adhesion strength of TiO_2 coating showed a decreased trend from 3.88 MPa to 2.05 MPa as annealed substrate temperature increased from room temperature to 400°C.

2. The oxide film thickness had an influence on the TiO_2 deposition process because thick oxide film inhibits formation of a new surface.

3. The main bonding mechanism is metallurgical bonding which is newly form substrate surface of annealed Al 1050 that oxide free to TiO_2 particle.

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