

Article

Analysis of Thermally Oxidized Surfaces of Additive Manufacturing Metal Powders Using Triboelectric Charging

Ali N. Alagha , Eileen Ross L. Espiritu , Emilio Galindo , Camila Gutiérrez, Pierre Hudon and Mathieu Brochu *

REGAL Aluminum Research Center, Department of Mining and Materials Engineering, McGill University, Montreal, QC H3A 0C5, Canada; ali.alagha@mail.mcgill.ca (A.N.A.); emilio.galindo@mail.mcgill.ca (E.G.)

* Correspondence: mathieu.brochu@mcgill.ca

Abstract

In powder-based additive manufacturing (AM), thermal exposure during processing can modify powder surface chemistry and promote oxide growth, even under inert atmospheres from residual oxygen contamination. This study investigates the effect of thermal oxidation on the surface chemistry and triboelectric charging behavior of three AM metal alloy powders: AlSi10Mg, 316L stainless steel (SS 316L), and Ti6Al4V. The work examines the evolution of the oxide layer during baking at 100 and 300 °C using triboelectric charging corroborated by X-ray photoelectron spectroscopy (XPS), diffuse-reflectance spectroscopy, and work-function measurements. The results show that heating modifies the surface oxide state of all powders, with changes dependent on the alloy composition and baking temperature. For AlSi10Mg, heating modified the Al₂O₃-rich surface oxide, with changes consistent with increased oxide ordering and γ -Al₂O₃-like characteristics, with the work function increasing from 4.34 ± 0.01 eV in the as-received (AR) condition to 4.92 ± 0.03 eV after baking at 300 °C. The corresponding triboelectric n -exponent increased from 0.91 to 1.38. For SS 316L, transformation of Cr(OH)₃ to Cr₂O₃ reduced triboelectric charge accumulation, while the oxygen concentration increased from 49.92 to 54.87 at.% and the work function decreased from 5.74 ± 0.02 to 5.28 ± 0.04 eV after baking at 300 °C. This reflected a drop in the n -exponent from 0.82 for AR to 0.73 at 300 °C. For Ti6Al4V, charging variations were associated with titanium oxide evolution and surface modifications consistent with rutile-related titanium oxide characteristics, with the work function increasing from 5.33 ± 0.01 to 5.44 ± 0.03 eV after baking at 300 °C. The corresponding triboelectric n -exponent increased from 0.49 to 0.52. Overall, triboelectric charging is a sensitive approach for detecting thermally driven surface oxide modifications in additive manufacturing powders.

Keywords: aluminum alloys; Ti6Al4V alloy; stainless steel; triboelectric charging; powder characterization; surface chemistry; oxidation



Academic Editor: Nicanor Cimpoesu

Received: 1 June 2026

Revised: 31 July 2026

Accepted: 1 August 2026

Published: 4 August 2026

Copyright: © 2026 by the authors.

Licensee MDPI, Basel, Switzerland.

This article is an open access article distributed under the terms and conditions of the [Creative Commons Attribution \(CC BY\)](https://creativecommons.org/licenses/by/4.0/) license.

1. Introduction

Metal powders used in laser powder bed fusion (LPBF) are repeatedly exposed to thermal cycling, residual oxygen, humidity, spatter, sieving, and handling during processing and reuse. These exposures can modify the surface state of the particles even when the particle size distribution and morphology remain nearly unchanged [1]. In LPBF, potential modifications are important as the quality of the repeatedly spread thin powder layer depends on powder properties such as flowability, continuity, uniformity, and packing density [2–7]. Surface changes such as oxide growth, hydroxylation, and contamination can therefore influence powder spreading, laser–powder interaction, melt-pool stability,

and defect formation in printed parts [1,8]. Recent studies have shown that conventional powder metrics alone are not sufficient to fully describe powder quality. Ilangovan et al. [8] investigated AlSi10Mg powder surface chemistry and showed that surface oxidation increases defect formation during LPBF. Similarly, Elambasseril et al. [9] studied Ti6Al4V powders using surface chemistry, morphology, particle size distribution, rheological measurements, and powder-bed layer density. They showed that common powder metrics such as apparent density, tap density, and angle of repose alone were not sufficient to predict part quality. These studies show that powder characterization should include both bulk powder properties and surface-sensitive measurements.

Powder reuse studies have also highlighted the importance of surface chemistry. Warner et al. [10] used X-ray photoelectron spectroscopy (XPS) to investigate reused AlSi7Mg powder and showed that the surface remained dominated by Al_2O_3 , while SiO_2 increased with reuse. Our previous work on reused AlSi10Mg powder showed that reuse modified the aluminum oxide layer and changed its interaction with moisture [11,12]. Raza et al. [13] also reported degradation of AlSi10Mg powder during LPBF reuse, including the formation of a thicker oxide layer mainly composed of Al_2O_3 with MgAl_2O_4 spinel at the outer surface. For SS 316L, Kazi et al. [1] used high-resolution SEM and XPS to show that reuse modifies the chemistry of the surface oxide layer. These studies confirm that reuse-related degradation is strongly linked to the surface oxide state. However, reuse combines several effects at the same time, including thermal exposure, oxidation, spatter interaction, and humidity exposure. As a result, the effect of temperature alone on powder surface chemistry remains difficult to isolate from reuse studies.

The selected powders in this work represent three widely used LPBF material systems with different native oxide chemistries. AlSi10Mg is commonly used in powder bed additive manufacturing because of its low density and good processability [8,14]. However, the surface oxide thickness, oxide composition, and hydroxylated species can affect powder behavior and part quality [13]. SS 316L is widely used because of its corrosion resistance and processability, but its chromium-rich surface oxide can still evolve during thermal exposure and reuse [1,15]. Ti6Al4V is used in aerospace and biomedical applications, where oxygen pickup and titanium oxide evolution must be controlled to ensure quality [2]. Therefore, AlSi10Mg, SS 316L, and Ti6Al4V provide suitable systems for comparing thermally induced changes in Al-based, Cr-based, and Ti-based surface oxides.

Surface-sensitive techniques such as X-ray photoelectron spectroscopy (XPS) and Auger spectroscopy can provide detailed information on oxide species and chemical states, but they are expensive, time-consuming, and require specialized expertise. Their use is therefore limited for routine industrial powder quality control. In contrast, triboelectric charging has recently been proposed as a rapid method to detect surface changes in AM powders. Galindo et al. [16] introduced a triboelectric charging-based method for assessing AM metal powder quality using several alloy systems, including commercially pure titanium (CpTi), Ti6Al4V, AlSi10Mg, IN738, and SS 316L. Their work showed that differences in powder surface chemistry identified by XPS can be reflected in the triboelectric charging response. This method was further applied to aluminum alloy powders, where changes in composition, particle size distribution, moisture adsorption, hydroxylated species, and surface oxide condition were associated with specific variations in charge accumulation [17,18]. These studies show that triboelectric charging is sensitive to powder surface chemistry, but the effect of controlled thermal oxidation on the charging behavior of AM metal powders has not yet been systematically documented.

The present work investigates whether triboelectric charging can be used as a surface-sensitive indicator of thermally induced oxidation in AM metal powders when particle size distribution and morphology remain nearly unchanged. AlSi10Mg, SS 316L, and Ti6Al4V

powders were baked in air at 100 and 300 °C as an accelerated treatment to isolate the effect of thermal oxidation on the powder surface. The resulting surface changes were characterized using XPS, optical absorption, work function measurements, and triboelectric charging. The oxide chemistry evolution and work function changes were linked with the measured charge response, showing that triboelectric charging can be used as a complementary quality-control tool for detecting surface degradation in AM powders.

2. Materials and Methods

The work utilizes commercial AlSi10Mg powder that was supplied by Equispheres Inc. (Ottawa, ON, Canada) and produced using the technique described in [19], SS 316L powder supplied by Renishaw (Wotton-under-Edge, UK) and produced by gas atomization, and Ti6Al4V powder supplied by AP&C (Advanced Powders & Coatings, Boisbriand, QC, Canada) produced using plasma atomization. The particle size distribution (PSD) was measured using a Microtrac MRB SYNC particle size analyzer (Microtrac MRB, Osaka, Japan). The specific surface area (SSA) was calculated from the 2D particle projections obtained during the PSD analysis. Powder morphology was examined using a Hitachi SU3500 scanning electron microscope (SEM; Hitachi, Tokyo, Japan). Powder flow rate and apparent density were determined using a Hall flowmeter funnel (HLL Landgraf Laborsysteme, Langenhagen, Germany) following ASTM B213-20 and ASTM B212-21, respectively [20,21]. SEM images were acquired using an accelerating voltage of 10 kV for AlSi10Mg and 15 kV for SS 316L and Ti6Al4V. To produce controlled surface oxidation, the powders were baked in air at 100 and 300 °C for 60 min using a Lindberg box furnace (Lindberg, Riverside, MI, USA). This baking treatment provides a controlled approach to induce and compare thermally driven surface oxidation across different alloy powders. The as-received (AR) powders were used as the reference conditions. The baking temperatures were selected to represent moderate thermal exposure that powder particles may experience during LPBF within the thermally affected region surrounding the printed part. The powder bed is typically operated from room temperature to 200 °C with steep temperature gradients generated during printing [22]. Therefore, powders located near the printed part can experience higher thermal exposure above the build temperature and below the melting temperature. The selected temperatures of 100 and 300 °C were therefore chosen to represent the upper range of the thermally affected surrounding powder. All physical property tests were performed on three independently prepared powder samples for each condition, and the results are reported as the mean $\pm$ standard deviation.

Surface chemical characterization was performed using a K-Alpha monochromatic X-ray photoelectron spectrometer (Thermo Fisher Scientific, East Grinstead, UK) equipped with an Al K α X-ray source (1486.6 eV, 0.834 nm). Analyses were carried out in a high-vacuum chamber of 10^{-8} Torr. The procedure reported by Buzukashvili et al. [12] was followed, where an elemental survey was acquired at a pass energy of 200 eV and high-resolution spectra were collected at a pass energy of 50 eV. To minimize surface charging effects, an electron charge gun was used. Depth profiling was performed using the standard monatomic Ar⁺ sputtering method of the instrument, with a nominal ion energy of 1.0 keV, a sputtering raster area of $1 \times 1 \text{ mm}^2$, and a reference-material sputtering-rate calibration of approximately 0.1 nm/s. Spectra were collected after cumulative sputtering (etching) times of 0, 10, 20, 30, and 40 s for AlSi10Mg and SS 316L, and 0, 5, 10, 15, and 20 s for Ti6Al4V. The spectra were calibrated using the C 1s peak for C–C bonding at 284.8 eV and processed using CasaXPS, version 2.3.27 (Casa Software Ltd., Teignmouth, UK). The peak position and full widths at half maximum (FWHM) of each fitted component are provided in the Supplementary Materials.

The work function of the powders was measured using Photoemission Yield Spectroscopy in Air (PYSA) with a Riken AC-2 system (Riken Keiki Co., Tokyo, Japan). The measurements were carried out by exposing the powder surface to ultraviolet (UV) photons with energies ranging from 4.0 to 6.5 eV in 0.05 eV increments, using a light intensity of 25 nW and a counting time of 30 s per step. The work function was determined from the photoemission threshold by extrapolating the onset of the photoemission yield curve to the baseline. To ensure repeatability, measurements were performed on three independently prepared powder samples for each condition.

The optical absorption response of the powders was evaluated by diffuse reflectance spectroscopy (DRS) using a PerkinElmer LAMBDA 750 ultraviolet–visible–near-infrared (UV–Vis–NIR) spectrophotometer (PerkinElmer, Inc., Shelton, CT, USA), equipped with an integrating-sphere accessory. Diffuse reflectance spectra were recorded over the wavelength range of 400–1300 nm using BaSO₄ powder as the reference material. The powders were prepared for measurements in a quartz-glass cuvette. At the LPBF laser wavelength, metals exhibit optical penetration depths in the order of nanometers. Therefore, transmission through the powder samples was assumed to be negligible [23]. With this assumption, the apparent absorption (%) was calculated as $[A(\%) = 100 - R(\%)]$, where (R) is the measured diffuse reflectance.

Triboelectric charging experiments were conducted using a GranuCharge™ apparatus (GranuTools, Awans, Belgium) within a controlled atmosphere chamber maintained at 25 °C and 30% relative humidity. The initial and final charge of the powder were measured by flowing the powder through a fixed stainless-steel V-tube geometry at different feeding times of 15, 20, 25, 30, and 35 s. The powder mass flow rate was measured using a balance positioned beneath the V-tube. For each feeding time of 15, 20, 25, 30, and 35 s, the mass of powder collected after passing through the tube was recorded as a function of time, and the average mass flow rate was calculated from the slope of the mass-versus-time curve. For each feeding time, the charging data were fitted by nonlinear regression using the following compressed exponential model [24]:

$$Q(t) = Q_f[1 - \exp(-(\alpha t)^\beta)], \quad (1)$$

where $Q(t)$ is the normalized charge density at time t (nC/m²), Q_f is the saturation charge density (nC/m²), t is the charging time (seconds), α is the characteristic charging-rate constant (s^{−1}), and β is a dimensionless parameter. The value of β was fixed at 1.5 for all powder conditions, consistent with the previously established fitting procedure of several metallic systems, including Al, Ti, Fe, and Ni-based materials [16]. Q_f and α were treated as free parameters and obtained by nonlinear least-squares fitting of Equation (1).

The fitted Q_f and α pairs obtained under the different feeding conditions were subsequently related using the empirical power-law expression:

$$|Q_f| = k\alpha^n \quad (2)$$

where k is the fitted proportionality coefficient and n is a dimensionless exponent describing the dependence of the charge density on the characteristic charging rate. Further details on the method are provided in [16–18].

3. Results and Discussion

3.1. AlSi10Mg Powder

The AlSi10Mg powder was first analyzed to determine whether baking modified the particle size distribution, morphology, and surface chemistry of the powder. The as-received (AR) powder was used as the reference condition, while the powders baked at 100

and 300 °C were used to evaluate the effect of thermal exposure. Table 1 shows the PSD main diameters, SSA, and Hall flow data of AlSi10Mg powders. The main particle size diameters (D_{10} , D_{50} and D_{90}), SSA, Hall flow rate and apparent density remained nearly unchanged for all conditions. These results indicate that the heat treatment produced minor variations in the measured extrinsic powder characteristics. Figure 1 presents SEM micrographs of the AR and baked powders. In all conditions, the particles remained predominantly spherical with smooth surfaces with some satellites.

Table 1. PSD, SSA and Hall flow data of the AlSi10Mg powders.

Powder Condition	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)	SSA (m^2/g)	Hall Flow Rate (g/s)	Apparent Density (g/cm^3)
AlSi10Mg, AR	43 ± 2	55 ± 3	74 ± 6	0.04	0.90 ± 0.01	1.573 ± 0.003
AlSi10Mg, 100 °C	42 ± 1	54 ± 1	73 ± 2	0.04	1.06 ± 0.02	1.536 ± 0.005
AlSi10Mg, 300 °C	42 ± 2	54 ± 1	72 ± 4	0.04	0.87 ± 0.03	1.469 ± 0.001

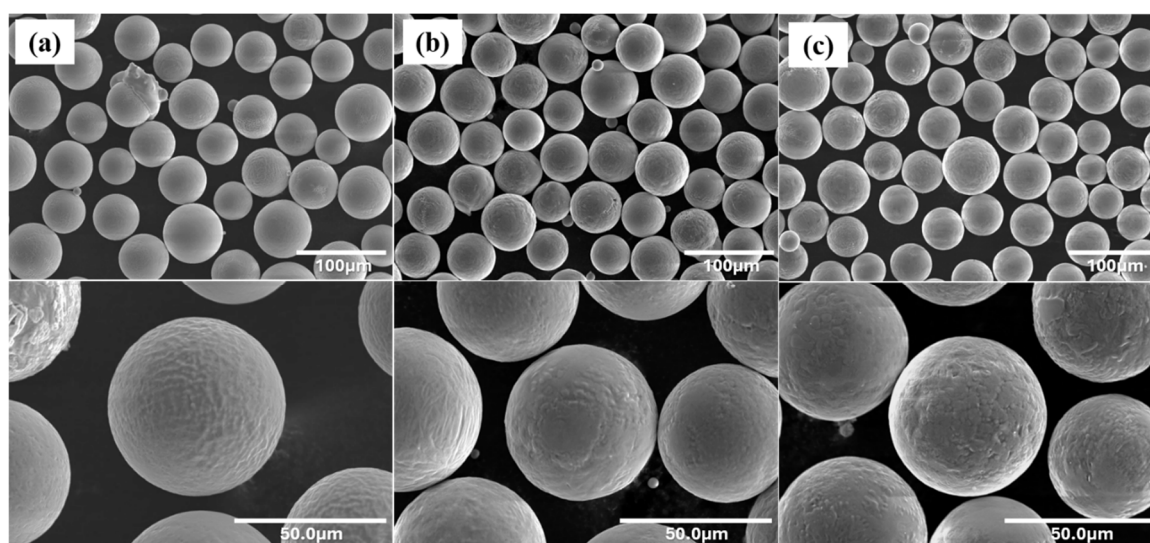


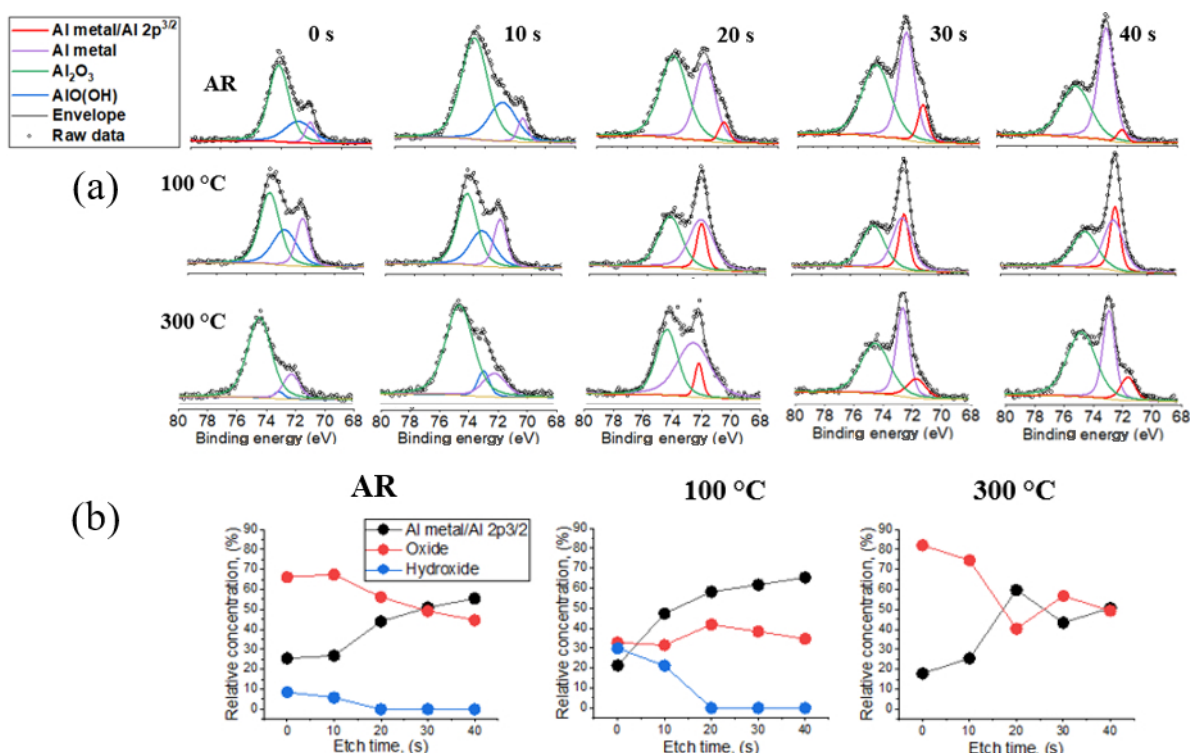
Figure 1. Low- and high-magnification SEM micrographs of (a) AR and baked at (b) 100 °C and (c) 300 °C AlSi10Mg powders.

Table 2 summarizes the atomic concentrations of Al 2p, O 1s, Mg 1s, Si 2p, and C 1s measured at different etching times for the AR and baked powders. For the AR powder, the Al 2p concentration increased from 15.19 at.% at the surface to 37.33 at.% after 40 s of etching, indicating a progressive thinning of the oxide layer with etching. At the same etching depth, the Al 2p concentration remained lower in all baked powders than in the AR condition, confirming that baking increased the oxide layer thickness. Overall, the XPS survey indicates progressive oxidation with increasing baking temperature.

Figure 2a shows the high-resolution Al 2p spectra to further clarify the changes in surface chemistry. Three main contributions were identified, corresponding to metallic Al (72.6 eV), Al_2O_3 (74.2 eV), and $\text{AlO}(\text{OH})$ (75.6 eV) [25]. The Al $2p^{3/2}$ peak was detected at 71.8 eV in some specific cases. Figure 2b depicts the relative concentrations of the species detected as a function of etching time. As the baking temperature increased, the metallic Al and $\text{AlO}(\text{OH})$ contributions progressively decreased. In particular, the absence of the $\text{AlO}(\text{OH})$ peak at shorter etching time for the 300 °C condition indicates conversion of hydroxylated species into Al_2O_3 [26]. In addition, the Al $2p^{3/2}$ component showed a progressive reduction at higher baking temperatures, further indicating modification of the oxide structure with heating [27].

Table 2. Surface concentration (at.%) of the AlSi10Mg powders from XPS survey.

AlSi10Mg, AR					
Etching Time, s	Al 2p	O 1s	Mg 1s	Si 2p	C 1s
0	15.19	47.26	6.07	4.22	27.27
10	24.84	48.12	11.35	5.66	10.04
20	28.88	42.46	9.88	8.66	10.10
30	33.62	33.07	8.05	11.14	9.82
40	37.33	33.79	5.99	11.62	11.25
AlSi10Mg, 100 °C					
Etching Time, s	Al 2p	O 1s	Mg 1s	Si 2p	C 1s
0	16.14	42.98	7.17	5.81	27.89
10	23.81	38.91	8.55	9.72	19.03
20	25.56	32.92	7.69	13.07	20.75
30	28.81	29.01	6.37	14.87	20.93
40	30.84	25.99	5.37	15.91	21.90
AlSi10Mg, 300 °C					
Etching Time, s	Al 2p	O 1s	Mg 1s	Si 2p	C 1s
0	11.65	46.38	11.00	3.38	27.60
10	15.98	45.22	21.08	5.02	12.71
20	18.13	43.08	19.35	6.49	12.95
30	21.37	40.39	16.84	7.51	13.89
40	25.19	37.26	14.17	9.53	13.86

**Figure 2.** (a) High-resolution Al 2p spectra and (b) relative content vs. sputtering (etching) time plots of the AR and baked powders.

To further support the interpretation of the temperature-induced surface changes in AlSi10Mg, additional Auger parameter (AP) analysis was performed following the Wagner approach [28–30]. The detailed results are provided in the Supplementary Materials. The observed Auger parameter shifts are consistent with changes in the structural and electronic

state of the Al_2O_3 -rich surface oxide and suggest increased oxide ordering after baking at 300 °C. Similar shifts have been associated in the literature with γ - Al_2O_3 -like oxide characteristics [27,31]. These shifts are in line with previous work linking Auger parameter variations to changes in oxide structure and defect state [11,28,30,32]. As suggested by Van den Brand et al. [33], shifts in the AP obtained by XPS analysis are associated with changes in the Fermi level position within the oxide band gap, which in the case of Al_2O_3 are induced by defect states due to an oxide enrichment. The changes observed in this work are therefore consistent with increased Al_2O_3 ordering or γ - Al_2O_3 formation. These transitions have also been reported in other work at oxidation temperatures of 100 and 200 °C [33–35].

Figure 3 shows the apparent optical absorption spectra of the AlSi10Mg powders between wavelengths of 400 and 1300 nm. At the LPBF laser wavelength of 1070 nm, the AR powder absorbed 65.5% of the incident light, compared with 66.1% for the 100 °C powder and 67.5% for the 300 °C powder. Relative to the AR condition, this corresponds to increases of 0.9% and 3.0% for the 100 and 300 °C powders, respectively. Based on the XPS and Auger parameter results, this increase in absorption is associated with modification and increased ordering of the Al_2O_3 -rich surface oxide at 300 °C, with characteristics consistent with γ - Al_2O_3 -like ordering.

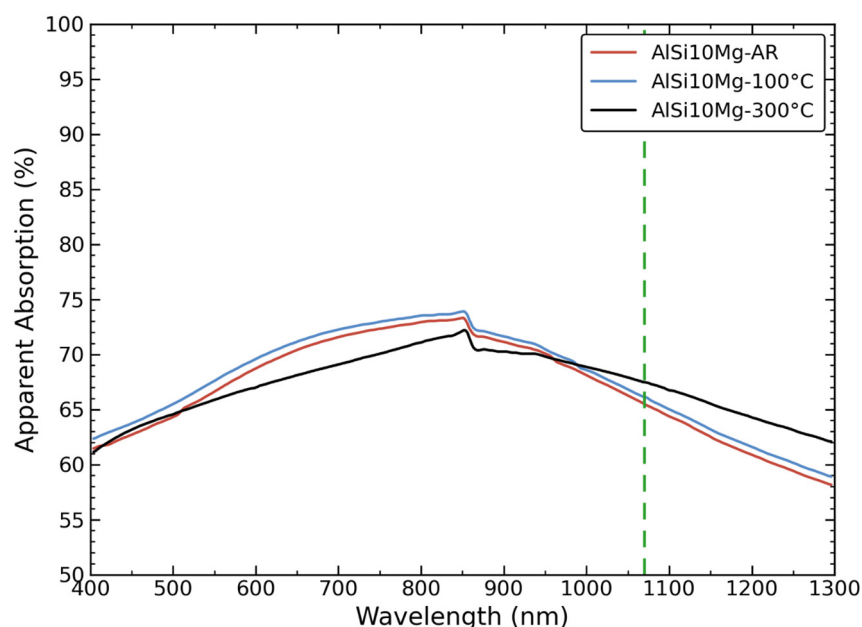


Figure 3. Apparent optical absorption as a function of wavelength for AlSi10Mg powders under different conditions. The vertical dashed line represents the wavelength of LPBF fiber laser ($\lambda = 1070$ nm).

Figure 4a shows the triboelectric charging behavior of the AlSi10Mg powders as a function of mass flow rate. As the baking temperature increased from the AR state to 300 °C, the powders accumulated increasingly negative charge. This behavior can be interpreted based on the literature-reported work function difference between the stainless-steel V-tube (4.08–4.19 eV) and the oxide-covered aluminum particles [36,37]. The work function of the powders, measured by PYSA, was 4.34 ± 0.01 eV for the AR powder, 4.20 ± 0.03 eV after baking at 100 °C, and increased to 4.92 ± 0.03 eV after baking at 300 °C. Since the stainless-steel tube has a lower work function than the oxide surface of the powder, electron transfer occurs toward the powder during triboelectric charging, producing a negative charge on the particles. The increase in negative charge with baking temperature is also consistent with the XPS results, which showed modification of the Al_2O_3 -rich surface layer and a reduction in hydroxylated aluminum species after baking. The more negative

charging observed from 100 to 300 °C is therefore consistent with the changes in the surface oxide identified by XPS.

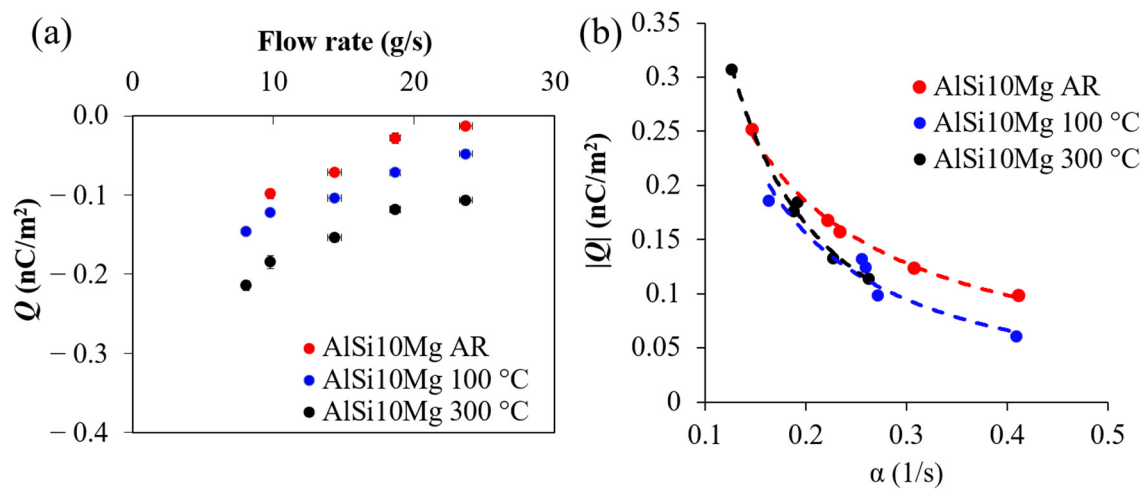


Figure 4. Triboelectric charging data; (a) accumulated charge density as a function of mass flow rate, and (b) empirical power equation fitting for AlSi10Mg powders.

Table 3 provides the n -exponent of the powders obtained from the fitting of Figure 4b. The fitted n -exponent showed a dependence on baking temperature. The AR powder exhibited an exponent of 0.91, which increased to 1.23 at 100 °C. This increase is consistent with thickening of the amorphous- Al_2O_3 layer and matches the calculated shift in the XPS analysis of Figure 2 [31]. From 100 to 300 °C, the exponent increased to 1.38, ascribed to an increase in the degree of crystallinity of the γ - Al_2O_3 and the continued structural ordering of the oxide [31,38].

Table 3. Surface properties of the AlSi10Mg powders and the fitted n -exponent.

Powder Condition	Surface State	n -exponent	R^2
AR	Amorphous- Al_2O_3	0.91 ± 0.01	0.99
AlSi10Mg, 100 °C	Amorphous- Al_2O_3 enrichment	1.23 ± 0.03	0.93
AlSi10Mg, 300 °C	γ - Al_2O_3	1.38 ± 0.01	0.99

3.2. SS 316L Powder

The PSD, SSA, and Hall flow data of the SS 316L powders are summarized in Table 4. Minor variations in the data were observed after baking. Figure 5 shows SEM micrographs of the AR and baked powders. In all conditions, the powder consisted of spherical, elongated, and slightly deformed particles, with some satellites. These observations indicate that baking produced minor variations in the extrinsic powder characteristics.

Table 4. PSD, SSA and Hall flow data of SS 316L powder at different conditions.

Powder	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)	SSA (m^2/g)	Hall Flow Rate (g/s)	Apparent Density (g/cm^3)
SS 316L, AR	25 ± 1	35 ± 4	50 ± 3	0.03	2.56 ± 0.03	4.322 ± 0.009
SS 316L, 100 °C	26 ± 3	36 ± 1	53 ± 1	0.03	2.49 ± 0.01	4.336 ± 0.010
SS 316L, 300 °C	22 ± 1	32 ± 2	50 ± 1	0.03	2.28 ± 0.06	4.335 ± 0.027

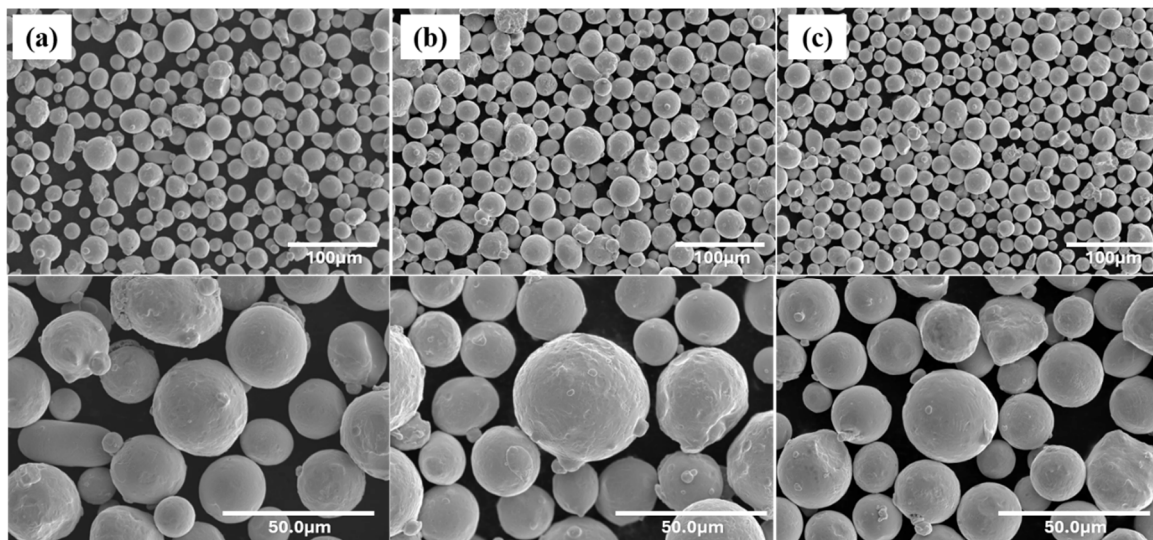


Figure 5. Low- and high-magnification SEM micrographs of (a) AR and baked at (b) 100 °C and (c) 300 °C SS 316L powders.

Table 5 summarizes the XPS survey atomic concentrations of Fe, Cr, Ni, O, and C measured on the surface of the SS 316L powders by XPS. The concentrations of Fe 2p and Ni 2s changed only slightly after baking at 100 and 300 °C compared with the AR powder. In contrast, the Cr 2p concentration increased more noticeably at 300 °C, reaching 3.36 at.%. The O 1s concentration also changed with baking, increasing to 53.06 at.% at 100 °C and 54.87 at.% at 300 °C. These variations suggest modification of the chromium-based oxide species present at the powder surface.

Table 5. Surface concentration (at.%) of SS 316L powders from XPS survey.

Sample	Fe 2p	Cr 2p	Ni 2s	O 1s	C 1s
SS 316L, AR	12.05	2.57	0.70	49.92	34.76
SS 316L, 100 °C	12.15	2.67	0.76	53.06	31.36
SS 316L, 300 °C	12.01	3.36	0.71	54.87	29.05

Figure 6 presents the high-resolution Cr 2p spectra and the corresponding depth profiles for the SS 316L powders. Three main peaks were identified and assigned to metallic Cr, Cr₂O₃, and Cr(OH)₃ at binding energies of 574.10, 576.80, and 577.10 eV, respectively [25]. The AR powder surface was rich in both Cr₂O₃ and Cr(OH)₃, with relative contents of 39% and 42%, respectively, while the metallic Cr contribution was 19%. After baking at 100 °C, the relative content of Cr₂O₃ increased to 45%, indicating progressive oxidation of the chromium-rich surface. When the powder was baked at 300 °C, the Cr₂O₃ contribution further increased (55%), consistent with continued thermally driven oxidation. Stainless-steel powders are known to form a native chromium oxide layer during atomization [39,40], and the growth of this layer with temperature has been reported to follow parabolic kinetics controlled by chromium and oxygen diffusion [41]. In addition, Li et al. [42] and Zhang et al. [15] reported that α-Cr₂O₃ can form in the temperature range between 100 °C and 200 °C and remain stable at higher temperatures. Therefore, the Cr₂O₃ enrichment observed after baking at 300 °C is consistent with the development of α-Cr₂O₃-related oxide characteristics.

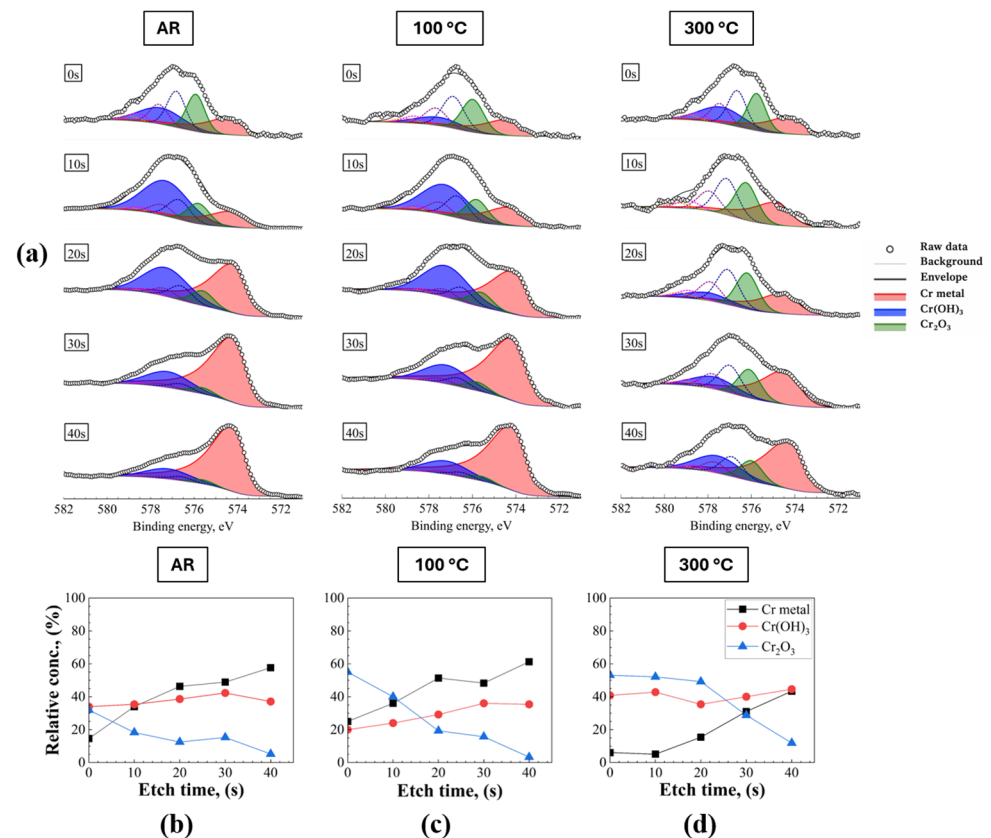


Figure 6. (a) High-resolution Cr 2p spectra and graphs of relative content vs. etch time of (b) AR, (c) baked at 100 °C and (d) 300 °C SS 316L powders.

Figure 7 presents the apparent absorption spectra of the SS 316L powders between wavelengths of 400 and 1300 nm. At the LPBF laser wavelength of 1070 nm, the AR powder absorbed 63.6% of the incident light, compared with 64.3% for the 100 °C powder and 64.0% for the 300 °C powder. Relative to the AR condition, this corresponds to increases of 1.1% and 0.7% for the 100 and 300 °C powders, respectively. Baking therefore produced a minimal increase in laser absorption of SS 316L in all conditions.

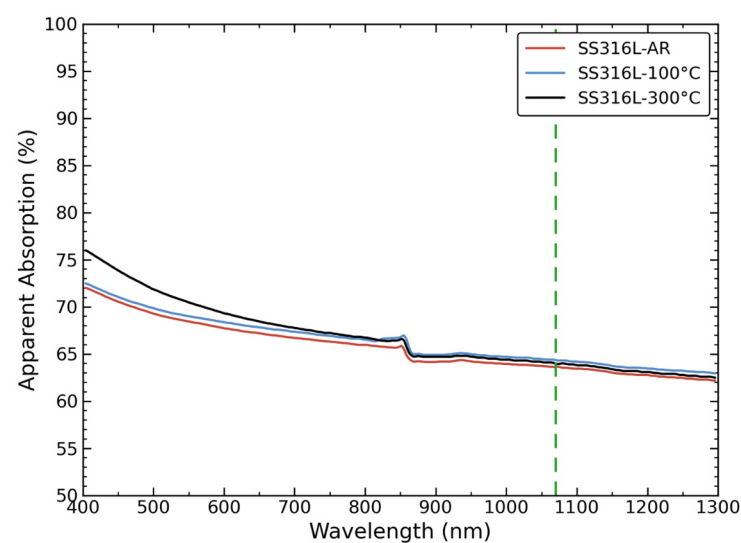


Figure 7. Apparent optical absorption as a function of wavelength for SS 316L powders at different conditions. The vertical dashed line represents the wavelength of LPBF fiber laser ($\lambda = 1070$ nm).

Figure 8 shows the triboelectric charging behavior of the SS 316L powders. For all conditions, the magnitude of the accumulated charge decreased as the flow rate increased, indicating that longer powder-tube interaction promoted greater charge accumulation. Since both the powder and the V-tube are stainless-steel-based systems, a nearly neutral charging response would be expected. However, charge accumulation can still occur due to differences in surface state between the powder and the tube, including differences in oxide coverage, crystallographic orientation, and surface contamination [43–45]. These factors modify the surface electron distribution and, consequently, the triboelectric response [46].

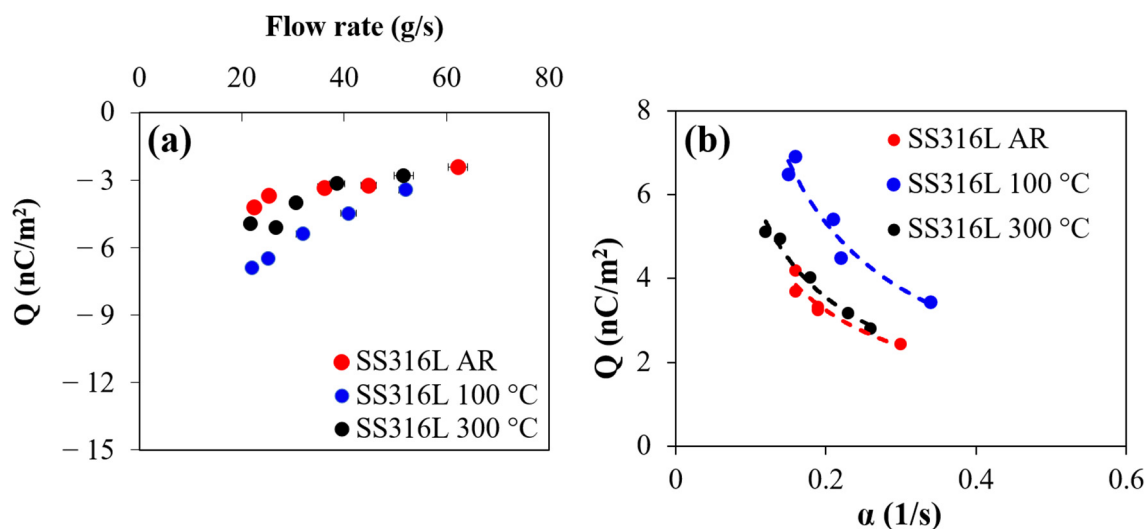


Figure 8. Triboelectric charging data; (a) accumulated charge density as a function of mass flow rate, and (b) empirical power equation fitting for SS 316L powders.

The work function measurements support the surface modifications identified by XPS. In particular, the work function of the SS 316L powder decreased from 5.74 ± 0.02 eV in the AR condition to 5.35 ± 0.01 eV after baking at 100 °C, and further to 5.28 ± 0.04 eV after baking at 300 °C. This reduction indicates that baking altered the electronic surface state of the powder, consistent with the evolution of chromium-based oxide species observed by XPS. For the AR powder, the charging behavior is therefore attributed to surface-state differences between the powder and the V-tube. After baking at 100 °C, the transformation of $\text{Cr}(\text{OH})_3$ into Cr_2O_3 modified both the oxide composition and the surface electronic state, which is reflected in the work function decrease and the corresponding triboelectric charging response. At 300 °C, the oxide layer evolved further, with additional enrichment and modification of the Cr_2O_3 -rich surface. These changes are consistent with α - Cr_2O_3 -related oxide characteristics [15], together with the continued decrease in work function and the distinct charging behavior measured for this condition. Thus, for SS 316L, the triboelectric response follows the changes in chromium oxide structure and the associated variation in work function induced by thermal oxidation [37].

Table 6 provides the n -exponent of the powders obtained from the fitting of Figure 8b. The fitted n -exponents for SS 316L were 0.82 for the AR powder, 0.87 for the powder baked at 100 °C, and 0.73 for the powder baked at 300 °C. These variations indicate that the kinetics of charge accumulation changed with baking temperature, in agreement with the oxide evolution identified by XPS and the work function measurements. In particular, the increase in n -exponent at 100 °C is associated with the initial transformation of hydroxylated chromium species, whereas the lower n -exponent at 300 °C is associated with further α - Cr_2O_3 -related ordering, resulting in charge dissipation.

Table 6. Surface properties of the SS 316L powders and the fitted n -exponent.

Powder Condition	Surface State	n -exponent	R^2
SS 316L, AR	Cr_2O_3 , $\text{Cr}(\text{OH})_3$	0.82 ± 0.02	0.94
SS 316L, 100 °C	Cr_2O_3	0.87 ± 0.01	0.94
SS 316L, 300 °C	$\alpha\text{-Cr}_2\text{O}_3$	0.73 ± 0.04	0.97

3.3. Ti6Al4V Powder

The PSD, SSA, and Hall flow data of the Ti6Al4V powders are reported in Table 7. Similar to the case of SS 316L and AlSi10Mg, minor differences were observed between the AR powder and the powders baked at 100 and 300 °C. Figure 9 shows SEM micrographs of the three conditions. The powders were predominantly spherical, with minor amounts of deformed particles and satellites. No substantial morphological change was observed after baking.

Table 7. PSD, SSA and Hall flow data of Ti6Al4V powder at different conditions.

Powder	D ₁₀ (μm)	D ₅₀ (μm)	D ₉₀ (μm)	SSA (m^2/g)	Hall Flow Rate (g/s)	Apparent Density (g/cm^3)
Ti6Al4V, AR	27 ± 2	36 ± 3	51 ± 5	0.05	1.22 ± 0.02	2.601 ± 0.007
Ti6Al4V, 100 °C	24 ± 2	33 ± 4	48 ± 3	0.05	1.10 ± 0.01	2.603 ± 0.004
Ti6Al4V, 300 °C	23 ± 1	32 ± 2	47 ± 6	0.05	1.12 ± 0.03	2.613 ± 0.002

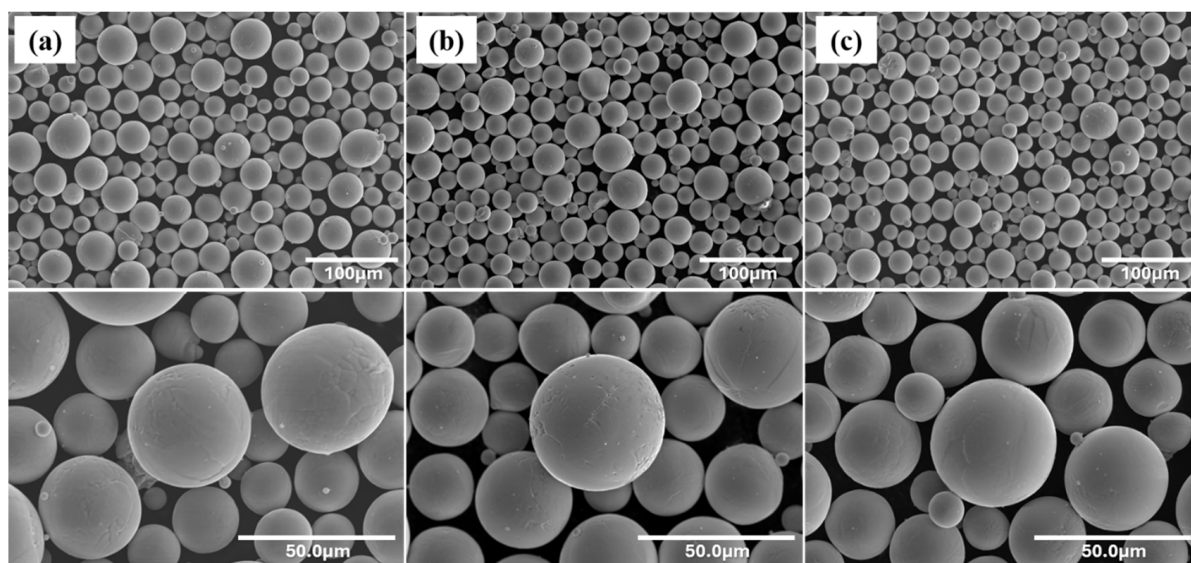
**Figure 9.** Low- and high-magnification SEM micrographs of Ti6Al4V powders (a) AR, (b) baked at 100 °C and (c) baked at 300 °C.

Table 8 summarizes the XPS survey atomic concentrations of Ti, Al, O, and C on the surface of the Ti6Al4V powders. The Ti 2p concentration varied between 13.18 and 15.01 at.%, which increased with baking temperature, while the Al 2p concentration remained nearly constant. The O 1s concentration increased by 1.3 at.% from the AR condition to the powder baked at 300 °C. These variations suggest modification of the titanium oxide species present at the surface after thermal exposure.

Table 8. XPS surface concentration (at.%) of the Ti6Al4V powders from XPS survey.

Sample	Ti 2p	Al 2p	O 1s	C 1s
Ti6Al4V, AR	13.18	8.46	50.40	27.96
Ti6Al4V, 100 °C	13.37	8.43	50.81	27.39
Ti6Al4V, 300 °C	15.01	8.53	51.70	24.76

Figure 10 presents the high-resolution Ti 2p spectra and the corresponding depth profiles for the Ti6Al4V powders. Peaks associated with Ti metal, Ti(II), Ti(III), and Ti(IV) were identified at binding energies of 454.0, 455.6, 456.8, and 458.5 eV, respectively [25]. The relative contributions of metallic Ti and Ti(III) remained relatively stable with etching depth, ranging from 15 to 25% for metallic Ti and from 5 to 20% for Ti(III). In contrast, Ti(IV) was the dominant species at the surface for all conditions, with a relative content of about 75% for the AR powder, increasing to 85% for the powder baked at 300 °C. Although the relative content of Ti(II) was minimal at the surface of the powder, its contribution increased with etching depth and reached up to 40%, while the relative content of Ti(IV) decreased to about 40%. Vaquila et al. [47] reported that oxidation of titanium between room temperature and 200 °C mainly produces Ti(IV), while oxidation in the range of 200 °C < T < 350 °C results in Ti(II) and Ti(III) species. In addition, the presence of Al and V in Ti6Al4V promotes the stabilization of rutile-related oxide structures during oxidation [48,49]. The surface changes observed for the powder baked at 300 °C are therefore consistent with rutile-related oxide characteristics.

Figure 11 shows the apparent absorption spectra of the Ti6Al4V powders between wavelengths of 400 and 1300 nm. At the LPBF laser wavelength of 1070 nm, the AR powder absorbed 67.5% of the incident light, compared with 68.2% for the 100 °C powder and 68.7% for the 300 °C powder. Relative to the AR condition, this corresponds to increases of 1.1% and 1.9% for the 100 and 300 °C powders, respectively. This behavior is consistent with the XPS results, which indicated the evolution of titanium oxide species and modification of the TiO₂-rich surface oxide at 300 °C, with characteristics consistent with rutile-related ordering, together with Ti(II) and Ti(III) suboxide species. This increase in absorption at the laser wavelength is attributed to the oxygen-deficient TiO_x species, which introduce electronic states within the band gap and have been reported to absorb in the NIR region [50].

The work function measurements further support this interpretation. The work function of Ti6Al4V was 5.33 ± 0.01 eV for the AR powder, remained nearly unchanged at 5.32 ± 0.02 eV after baking at 100 °C, and then increased to 5.44 ± 0.03 eV after baking at 300 °C. The negligible change at 100 °C suggests that the oxide layer remained dominated by Ti(IV)-rich species with limited structural modification, which is consistent with the lower charge accumulation measured for this condition. In contrast, the increase in work function at 300 °C indicates a modification of the oxide surface, with characteristics that are consistent with rutile-related oxide ordering, together with the appearance of additional titanium oxide species identified by XPS. This change in the surface electronic state matches the increase in charge accumulation observed for the powder baked at 300 °C. Therefore, for Ti6Al4V, the combined XPS and work function results indicate that the triboelectric behavior is linked to the thermal evolution of the titanium oxide layer.

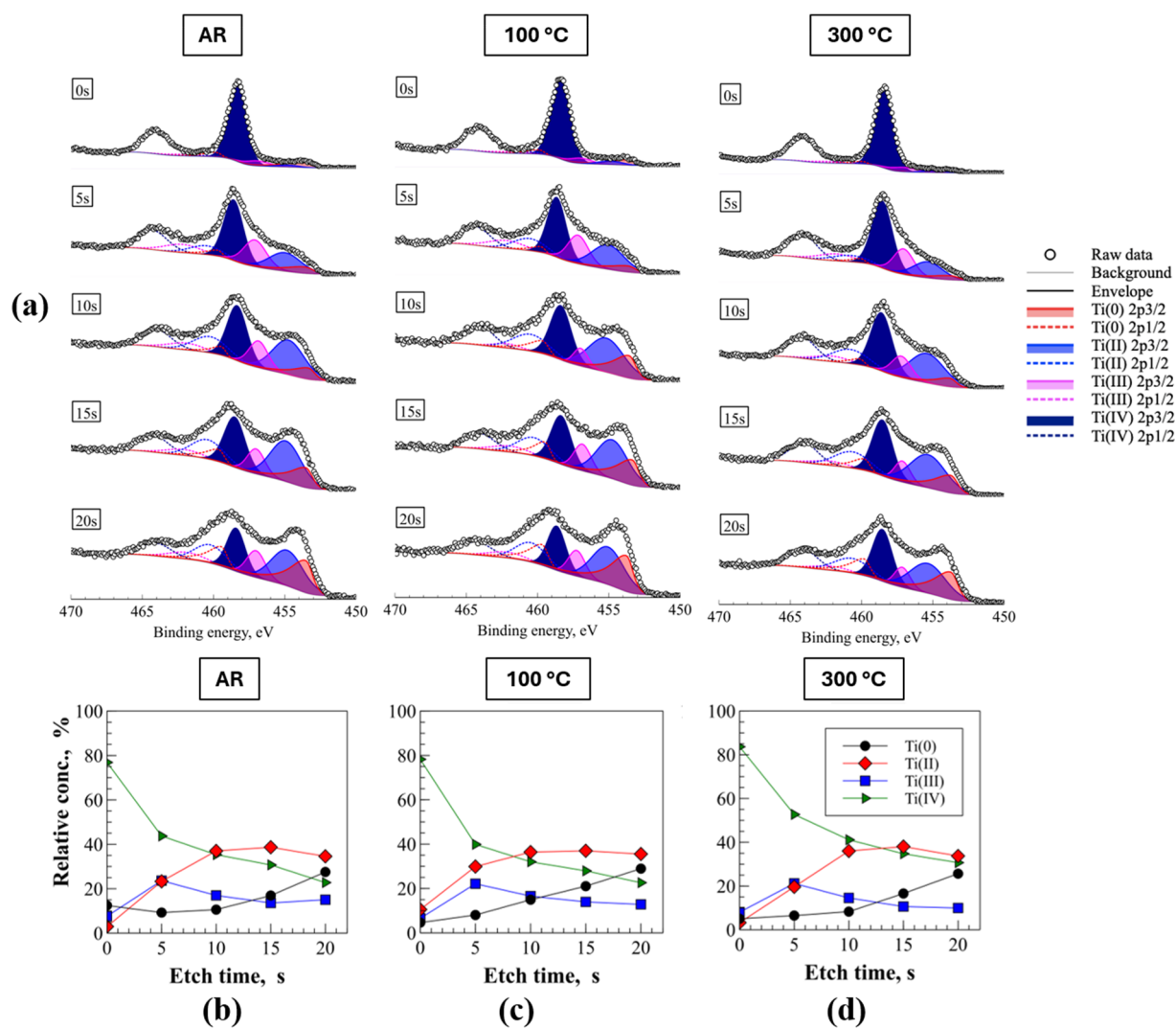


Figure 10. (a) High-resolution Ti 2p spectra and graphs of relative content vs. sputtering (etching) time of (b) AR, (c) baked at 100 °C and (d) 300 °C Ti6Al4V powders.

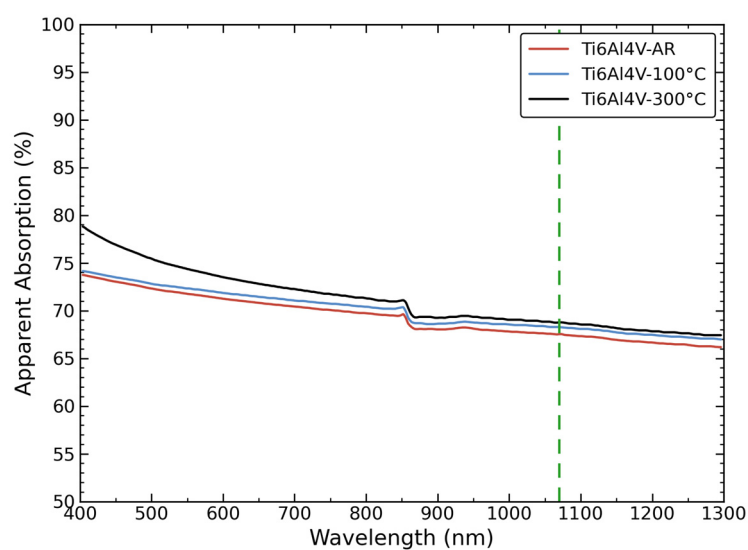


Figure 11. Apparent optical absorption as a function of wavelength for Ti6Al4V powders under different conditions. The vertical dashed line represents the wavelength of LPBF fiber laser ($\lambda = 1070$ nm).

Figure 12a shows the triboelectric charging behavior of the Ti6Al4V powders as a function of flow rate. The variation in charging response with temperature indicates that thermal oxidation altered the oxide structure and therefore the charge transfer behavior of the powder.

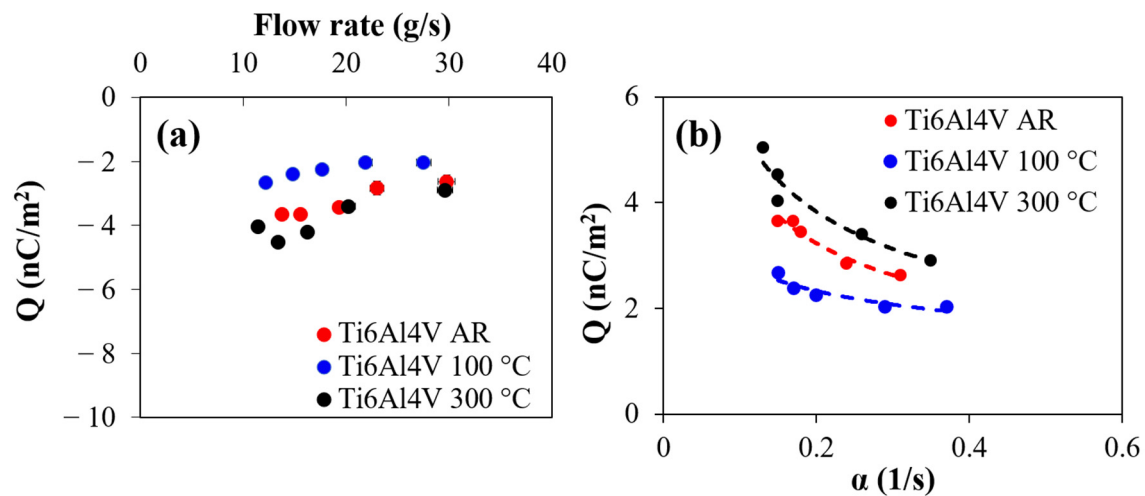


Figure 12. Triboelectric charging data; (a) accumulated charge density as a function of mass flow rate, and (b) empirical power equation fitting for the Ti6Al4V powders.

The Ti6Al4V charging data were analyzed using the compressed exponential model and power-law relationship described above, as provided in Figure 12b. The fitting n -exponent, corresponding to different surface species at the surface of the Ti6Al4V powder, is provided in Table 9. The n -exponents were 0.49 for the AR powder, 0.36 for the powder baked at 100 °C, and 0.52 for the powder baked at 300 °C. These variations in n -exponent are associated with the different titanium oxide species present at the surface after baking. Since the model is derived from a capacitor-based description of the charging process, the change in n -exponent is interpreted as reflecting variations in differential capacitance and charge relaxation behavior caused by the oxide structure present on the powder surface [51,52].

Table 9. Surface properties of the Ti6Al4V powders and the fitted n -exponent.

Powder Condition	Surface State	n -exponent	R^2
Ti6Al4V, AR	TiO ₂ , TiO	0.49 ± 0.02	0.95
Ti6Al4V, 100 °C	TiO ₂ , TiO	0.36 ± 0.01	0.92
Ti6Al4V, 300 °C	TiO ₂ /rutile-related species	0.52 ± 0.02	0.93

Overall, the combined work function and triboelectric charging results show that the charging response of AlSi10Mg, SS 316L, and Ti6Al4V powders is highly sensitive to thermally induced surface modifications. The oxide species formed after baking altered both the electronic surface state and the kinetics of charge accumulation, and these effects were captured by work function measurements and the fitted triboelectric charging n -exponents.

4. Conclusions

This study demonstrated that triboelectric charging can be used to assess surface chemistry changes induced by controlled thermal oxidation in AlSi10Mg, SS 316L, and Ti6Al4V powders, three material systems widely used in AM. In all powders, baking at 100 and 300 °C produced minor variations in the particle size distribution and morphology,

indicating that the changes in triboelectric charging behavior were mainly associated with thermally induced modifications in the surface oxide chemistry rather than changes in extrinsic powder characteristics. For AlSi10Mg, baking modified the Al₂O₃-rich surface layer and reduced hydroxylated aluminum species. The work function increased from 4.34 ± 0.01 eV in the AR condition to 4.92 ± 0.03 eV after baking at 300 °C, while the fitted *n*-exponent increased from 0.91 to 1.38. These changes indicate that thermal exposure modified both the oxide chemistry and the charge accumulation behavior of the powder. For SS 316L, baking promoted the evolution of chromium-based oxide species, with the oxygen concentration increasing from 49.92 to 54.87 at.% and the work function decreasing from 5.74 ± 0.02 to 5.28 ± 0.04 eV after baking at 300 °C. This resulted in a drop in the *n*-exponent from 0.82 for AR to 0.73 at 300 °C. For Ti6Al4V, the charging behavior was associated with the evolution of titanium oxide species, with the work function increasing from 5.33 ± 0.01 to 5.44 ± 0.03 eV after baking at 300 °C. The corresponding triboelectric *n*-exponent increased from 0.49 for AR to 0.52 at 300 °C. The change in triboelectric response indicates that the method is sensitive to thermally induced modifications of Ti-based surface oxides. The variation in the *n*-exponent of the fitting model with baking temperature indicated that the kinetics of charge accumulation depend on the nature and structure of the surface oxide layer. The main contribution of this work is the correlation between XPS-identified oxide evolution, work function variation, and triboelectric charging behavior across three AM powder systems with different oxide chemistries and their evolution with heating. Since the powders were oxidized in air, the treatment represents an accelerated comparative condition and does not directly reproduce the low-oxygen atmosphere or thermal history of LPBF. The results demonstrate the sensitivity of triboelectric charging to surface-oxide modifications and highlight its potential as a rapid complementary tool for powder-quality control.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/app16157778/s1>, Table S1. Nominal chemical composition of the AlSi10Mg powder as provided by the manufacturer; Table S2. Nominal chemical composition of the SS 316L powder as provided by the manufacturer; Table S3. Nominal chemical composition of the Ti6Al4V powder as provided by the manufacturer; Table S4. XPS Auger parameters of AlSi10Mg powders under different conditions; Table S5. XPS peak-fitting parameters for the Al 2p spectra of AlSi10Mg powders; Table S6. XPS peak-fitting parameters for the Cr 2p spectra of SS 316L powders; Table S7. XPS peak-fitting parameters for the Ti 2p spectra of Ti6Al4V powders; Table S8. Triboelectric charging data for AlSi10Mg powders; Table S9. Triboelectric charging data for SS 316L powders; Table S10. Triboelectric charging data for Ti6Al4V powders.

Author Contributions: Conceptualization, A.N.A., E.R.L.E., E.G., C.G. and M.B.; Methodology, A.N.A., E.R.L.E., E.G., P.H. and M.B.; Validation, A.N.A.; Formal analysis, A.N.A., E.G. and M.B.; Investigation, A.N.A., E.G., C.G. and M.B.; Resources, P.H. and M.B.; Data curation, A.N.A., P.H. and M.B.; Writing—original draft, E.R.L.E.; Writing—review & editing, A.N.A., E.G. and M.B.; Visualization, A.N.A., C.G. and M.B.; Supervision, M.B.; Project administration, M.B.; Funding acquisition, M.B. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Natural Sciences and Engineering Research Council of Canada (NSERC-ALLRP-5809).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article/Supplementary Material. Further inquiries can be directed to the corresponding author.

Acknowledgments: The authors would like to thank Gwénaél Chamoulaud of the Université du Québec à Montréal (UQAM) for his assistance with the UV–Vis–NIR measurements. The authors acknowledge the support of the Fonds de recherche du Québec—Nature et technologies (FRQNT) and the McGill Engineering Doctoral Award (MEDA) awarded to Ali Alagha.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Kazi, S.; Leach, L.; Hryha, E. Changes in Powder Surface Chemistry During Reuse of 316L in Powder Bed Fusion—Laser Beam. In *Proceedings of the Euro PM2024 Congress & Exhibition*; EPMA: Malmö, Sweden, 2024. [\[CrossRef\]](#)
2. Ni, C.; Zhu, J.; Zhang, B.; An, K.; Wang, Y.; Liu, D.; Lu, W.; Zhu, L.; Liu, C. Recent Advance in Laser Powder Bed Fusion of Ti–6Al–4V Alloys: Microstructure, Mechanical Properties and Machinability. *Virtual Phys. Prototyp.* **2025**, *20*, e2446952. [\[CrossRef\]](#)
3. Sun, X.; Chen, M.; Liu, T.; Zhang, K.; Wei, H.; Zhu, Z.; Liao, W. Characterization, Preparation, and Reuse of Metallic Powders for Laser Powder Bed Fusion: A Review. *Int. J. Extrem. Manuf.* **2024**, *6*, 012003. [\[CrossRef\]](#)
4. Chen, H.; Chen, Y.; Liu, Y.; Wei, Q.; Shi, Y.; Yan, W. Packing Quality of Powder Layer during Counter-Rolling-Type Powder Spreading Process in Additive Manufacturing. *Int. J. Mach. Tools Manuf.* **2020**, *153*, 103553. [\[CrossRef\]](#)
5. Vock, S.; Klöden, B.; Kirchner, A.; Weißgärber, T.; Kieback, B. Powders for Powder Bed Fusion: A Review. *Prog. Addit. Manuf.* **2019**, *4*, 383–397. [\[CrossRef\]](#)
6. Leung, C.L.A.; Marussi, S.; Towrie, M.; Atwood, R.C.; Withers, P.J.; Lee, P.D. The Effect of Powder Oxidation on Defect Formation in Laser Additive Manufacturing. *Acta Mater.* **2019**, *166*, 294–305. [\[CrossRef\]](#)
7. Mani, M.; Lane, B.; Donmez, A.; Feng, S.; Moylan, S.; Fesperman, R. *Measurement Science Needs for Real-Time Control of Additive Manufacturing Powder Bed Fusion Processes*; NIST Interagency/Internal Report (NISTIR) 8036; National Institute of Standards and Technology: Gaithersburg, MD, USA, 2015. [\[CrossRef\]](#)
8. Ilangovan, R.K.; Koundinya, N.T.B.N.; Krishnaswamy, H.; Saravana Kumar, G.; Amirthalingam, M.; Kottada, R.S. Influence of Powder Surface Chemistry on the Defect Formation in AlSi10Mg Alloy Processed via Laser Powder Bed Fusion Additive Manufacturing. *Materialia* **2024**, *34*, 102102. [\[CrossRef\]](#)
9. Elambasseril, J.; Song, T.; Mendis, S.; Lui, E.; Leary, M.; Brandt, M.; Qian, M. Effect of Powder Characteristics on Layer Density, Defects, and Tensile Properties of Ti–6Al–4V via Laser Powder Bed Fusion: Establishing Benchmark Parameters for Quality. *Prog. Addit. Manuf.* **2025**, *10*, 7449–7470. [\[CrossRef\]](#)
10. Warner, J.H.; Ringer, S.P.; Proust, G. The Influence of Powder Reuse on AlSi7Mg Manufactured by Laser Powder Bed Fusion. *Prog. Addit. Manuf.* **2026**, *11*, 6191–6208. [\[CrossRef\]](#)
11. Alagha, A.N.; Galindo, E.; Muñiz, J.; Espiritu, E.R.; Conlon, M.; Azari, K.; Butler-Jones, E.; Brochu, M. Detection of Surface Oxide Transformation during LPBF Re-Use of AlSi10Mg Powder via Triboelectric Charging and Its Effect on Moisture Interaction. *Adv. Powder Technol.* **2026**, *37*, 105254. [\[CrossRef\]](#)
12. Muñiz-Lerma, J.A.; Espiritu, E.R.L.; Butler-Jones, E.; Brochu, M.; Azari, K.; Conlon, M.J.; Nixon, G.; Valenti, J. Assessing the Influence of Aluminum Powder Characteristics on Reusability in Laser Powder Bed Fusion. *Prog. Addit. Manuf.* **2025**, *10*, 7685–7704. [\[CrossRef\]](#)
13. Raza, A.; Fiegl, T.; Hanif, I.; Markström, A.; Franke, M.; Körner, C.; Hryha, E. Degradation of AlSi10Mg Powder during Laser Based Powder Bed Fusion Processing. *Mater. Des.* **2021**, *198*, 109358. [\[CrossRef\]](#)
14. Martucci, A.; Aversa, A.; Lombardi, M. Ongoing Challenges of Laser-Based Powder Bed Fusion Processing of Al Alloys and Potential Solutions from the Literature—A Review. *Materials* **2023**, *16*, 1084. [\[CrossRef\]](#) [\[PubMed\]](#)
15. Zhang, L.; Kuhn, M.; Diebold, U. Growth, Structure and Thermal Properties of Chromium Oxide Films on Pt(111). *Surf. Sci.* **1997**, *375*, 1–12. [\[CrossRef\]](#)
16. Galindo, E.; Espiritu, E.R.L.; Gutierrez, C.; Alagha, A.N.; Hudon, P.; Brochu, M. A Method to Assess the Quality of Additive Manufacturing Metal Powders Using the Triboelectric Charging Concept. *Sci. Rep.* **2024**, *14*, 16439. [\[CrossRef\]](#) [\[PubMed\]](#)
17. Alagha, A.N.; Rossier, M.; Galindo, E.; Conlon, M.; Muñiz, J.; Azari, K.; Butler-Jones, E.; Brochu, M. Surface Chemistry Characterization of AA2014 Aluminum Alloy Powder through Triboelectric Charging. *Powder Technol.* **2025**, *449*, 120411. [\[CrossRef\]](#)
18. Alagha, A.N.; Galindo, E.; Conlon, M.; Muñiz, J.; Azari, K.; Butler-Jones, E.; Brochu, M. Surface Chemistry Analysis of Aluminum Powders through Triboelectric Charging: Effect of Composition, Particle Size Distribution and Moisture Adsorption. *Powder Technol.* **2025**, *468*, 121636. [\[CrossRef\]](#)
19. Conlon, M.J.; Phillips, J.; Dautre, S.R.; Azari Dorcheh, K.; Hafiz, A.M.K.; Bastien, J.; Johnson, K.G. Metal Powder-Based Manufacturing Process in Low Impurity Gas Atmosphere and System. U.S. Patent US12157169B2, 3 December 2024.
20. ASTM B213-20; Standard Test Methods for Flow Rate of Metal Powders Using the Hall Flowmeter Funnel. ASTM International: West Conshohocken, PA, USA, 2020. [\[CrossRef\]](#)

21. ASTM B212-21; Standard Test Method for Apparent Density of Free-Flowing Metal Powders Using the Hall Flowmeter Funnel. ASTM International: West Conshohocken, PA, USA, 2021. [\[CrossRef\]](#)
22. Bayoumy, D.; Boll, T.; Karapuzha, A.S.; Wu, X.; Zhu, Y.; Huang, A. Effective Platform Heating for Laser Powder Bed Fusion of an Al-Mn-Sc-Based Alloy. *Materials* **2023**, *16*, 7586. [\[CrossRef\]](#) [\[PubMed\]](#)
23. Tolochko, N.K.; Laoui, T.; Khlopkov, Y.V.; Mozzharov, S.E.; Titov, V.I.; Ignatiev, M.B. Absorptance of Powder Materials Suitable for Laser Sintering. *Rapid Prototyp. J.* **2000**, *6*, 155–161. [\[CrossRef\]](#)
24. Greason, W.D. Investigation of a Test Methodology for Triboelectrification. *J. Electrostat.* **2000**, *49*, 245–256. [\[CrossRef\]](#)
25. Wagner, C.D. *NIST X-Ray Photoelectron Spectroscopy (XPS) Database*; National Institute of Standards and Technology: Gaithersburg, MD, USA, 1990.
26. Jeurgens, L.P.H.; Sloof, W.G.; Tichelaar, F.D.; Mittemeijer, E.J. Growth Kinetics and Mechanisms of Aluminum-Oxide Films Formed by Thermal Oxidation of Aluminum. *J. Appl. Phys.* **2002**, *92*, 1649–1656. [\[CrossRef\]](#)
27. Snijders, P.C.C.; Jeurgens, L.P.H.P.H.; Sloof, W.G.G. Structural Ordering of Ultra-Thin, Amorphous Aluminium-Oxide Films. *Surf. Sci.* **2005**, *589*, 98–105. [\[CrossRef\]](#)
28. Wagner, C.D. Chemical Shifts of Auger Lines, and the Auger Parameter. *Faraday Discuss. Chem. Soc.* **1975**, *60*, 291. [\[CrossRef\]](#)
29. Wagner, C.D.; Joshi, A. The Auger Parameter, Its Utility and Advantages: A Review. *J. Electron Spectros. Relat. Phenom.* **1988**, *47*, 283–313. [\[CrossRef\]](#)
30. Moretti, G. The Wagner Plot and the Auger Parameter as Tools to Separate Initial- and Final-State Contributions in X-Ray Photoemission Spectroscopy. *Surf. Sci.* **2013**, *618*, 3–11. [\[CrossRef\]](#)
31. Jeurgens, L.P.H.; Sloof, W.G.; Tichelaar, F.D.; Mittemeijer, E.J. Structure and Morphology of Aluminium-Oxide Films Formed by Thermal Oxidation of Aluminium. *Thin Solid Films* **2002**, *418*, 89–101. [\[CrossRef\]](#)
32. Wagner, C.D.; Passoja, D.E.; Hillery, H.F.; Kinisky, T.G.; Six, H.A.; Jansen, W.T.; Taylor, J.A. Auger and Photoelectron Line Energy Relationships in Aluminum-Oxygen and Silicon-Oxygen Compounds. *J. Vac. Sci. Technol.* **1982**, *21*, 933–944. [\[CrossRef\]](#)
33. van den Brand, J.; Snijders, P.C.; Sloof, W.G.; Terryn, H.; de Wit, J.H.W. Acid-Base Characterization of Aluminum Oxide Surfaces with XPS. *J. Phys. Chem. B* **2004**, *108*, 6017–6024. [\[CrossRef\]](#)
34. McHale, J.M.; Navrotsky, A.; Perrotta, A.J. Effects of Increased Surface Area and Chemisorbed H₂O on the Relative Stability of Nanocrystalline γ -Al₂O₃ and α -Al₂O₃. *J. Phys. Chem. B* **1997**, *101*, 603–613. [\[CrossRef\]](#)
35. Castro, R.H.R.; Quach, D.V. Analysis of Anhydrous and Hydrated Surface Energies of Gamma-Al₂O₃ by Water Adsorption Microcalorimetry. *J. Phys. Chem. C* **2012**, *116*, 24726–24733. [\[CrossRef\]](#)
36. Marlow, F.; Josten, S.; Leiting, S. Electronics with Stainless Steel: The Work Functions. *J. Appl. Phys.* **2023**, *133*, 085104. [\[CrossRef\]](#)
37. Oje, A.M.; Ogwu, A.A.; Oje, A.I. An Investigation of the Work Function and Stability of Chromium Oxide Thin Films Deposited by Reactive Magnetron Sputtering. *J. Appl. Electrochem.* **2022**, *52*, 1551–1562. [\[CrossRef\]](#)
38. Trunov, M.A.; Schoenitz, M.; Zhu, X.; Dreizin, E.L. Effect of Polymorphic Phase Transformations in Al₂O₃ Film on Oxidation Kinetics of Aluminum Powders. *Combust. Flame* **2005**, *140*, 310–318. [\[CrossRef\]](#)
39. Norell, M.; Nyborg, L.; Tunberg, T.; Olefjord, I. Thickness Determination of Surface Oxides on Metal Powder by AES Depth Profiling. *Surf. Interface Anal.* **1992**, *19*, 71–76. [\[CrossRef\]](#)
40. Suri, P.; Koseski, R.P.; German, R.M. Microstructural Evolution of Injection Molded Gas- and Water-Atomized 316L Stainless Steel Powder during Sintering. *Mater. Sci. Eng. A* **2005**, *402*, 341–348. [\[CrossRef\]](#)
41. Tarabay, J.; Peres, V.; Pijolat, M. Oxidation of Stainless Steel Powder. *Oxid. Met.* **2013**, *80*, 311–322. [\[CrossRef\]](#)
42. Li, L.; Zhu, Z.; Yao, X.; Lu, G.; Yan, Z. Synthesis and Characterization of Chromium Oxide Nanocrystals via Solid Thermal Decomposition at Low Temperature. *Microporous Mesoporous Mater.* **2008**, *112*, 621–626. [\[CrossRef\]](#)
43. Kwetkus, B.A.; Sattler, K. Contact Charging of Oxidized Metal Powders. *Z. Phys. B Condens. Matter* **1991**, *82*, 87–93. [\[CrossRef\]](#)
44. Lacks, D.J.; Shinbrot, T. Long-Standing and Unresolved Issues in Triboelectric Charging. *Nat. Rev. Chem.* **2019**, *3*, 465–476. [\[CrossRef\]](#)
45. Matsusaka, S.; Maruyama, H.; Matsuyama, T.; Ghadiri, M. Triboelectric Charging of Powders: A Review. *Chem. Eng. Sci.* **2010**, *65*, 5781–5807. [\[CrossRef\]](#)
46. Ashcroft, N.W.; Mermin, N.D. *Solid State Physics*; Holt, Rinehart and Winston: New York, NY, USA, 1976.
47. Vaquila, I.; Passeggi, M.C.G.; Ferrón, J. Temperature Effects in the Early Stages of Titanium Oxidation. *Appl. Surf. Sci.* **1996**, *93*, 247–253. [\[CrossRef\]](#)
48. Depero, L.E.; Bonzi, P.; Zocchi, M.; Casale, C.; De Michele, G. Study of the Anatase-Rutile Transformation in TiO₂ Powders Obtained by Laser-Induced Synthesis. *J. Mater. Res.* **1993**, *8*, 2709–2715. [\[CrossRef\]](#)
49. Rinner, M.; Gerlach, J.; Ensinger, W. Formation of Titanium Oxide Films on Titanium and Ti6Al4V by O₂-Plasma Immersion Ion Implantation. *Surf. Coat. Technol.* **2000**, *132*, 111–116. [\[CrossRef\]](#)
50. Barros, H.W.S.; Duarte, D.A.; Sagás, J.C. Optical and Electrical Properties of Ti Suboxides Grown by Reactive Grid-Assisted Magnetron Sputtering. *Thin Solid Films* **2020**, *696*, 137762. [\[CrossRef\]](#)

51. Wintle, H.J. Surface-Charge Decay in Insulators with Nonconstant Mobility and with Deep Trapping. *J. Appl. Phys.* **1972**, *43*, 2927–2930. [[CrossRef](#)]
52. Macdonald, J.; Brachman, M. The Charging and Discharging of Nonlinear Capacitors. *Proc. IRE* **1955**, *43*, 71–78. [[CrossRef](#)]

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.